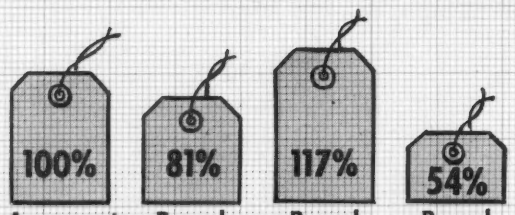
Corrosion

Official Publication
NATIONAL ASSOCIATION OF CORROSION ENGINEERS

Vol. 13 JULY, 1957 No. 7

YOU CAN'T KNOW
what the real price tag is...

How price per gallon compares on leading vinyls



UNLESS YOU
compare mil sq. ft. per gallon and...

Sq. ft. coverage per gallon, 1 mil thick

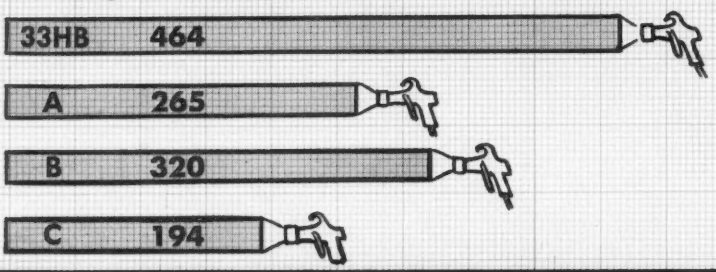
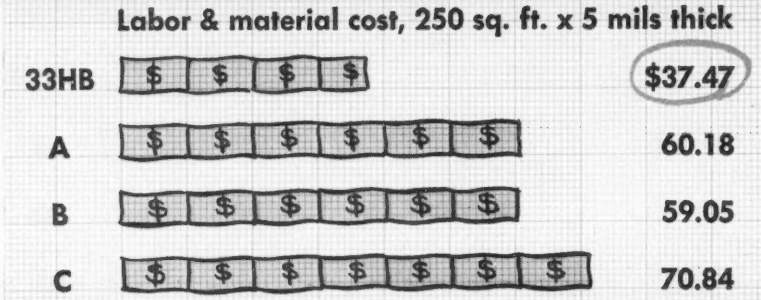


FIGURE THE
material cost of each coating, then get the...

		GALS.	COST
Needed to coat 250 sq. ft. 5 mils thick	33HB	2.68	$17.37
	A	4.72	24.78
	B	3.91	29.72
	C	6.44	22.54

REAL COST
by adding labor for the number of coats needed.

Labor & material cost, 250 sq. ft. x 5 mils thick



SERIES 102

Announcing... A True High Build Vinyl Coating

Amercoat 33HB now gives you even *more* "mils per gallon" —and at the lowest cost of any leading vinyl!

At last, the two major limitations of vinyl protective coatings—critical adhesion and low film thickness—have been largely overcome. Amercoat 86 Primer stopped adhesion troubles, eliminated underfilm corrosion and undercutting, and simplified surface preparation. *Now Amercoat 33HB Topcoat builds thick films in fewer coats at surprising savings.*

Amercoat 33HB is a "high-build" version of Amercoat 33, the standard of the protective coatings industry for over ten years. 33HB gives you the same high resistance to corrosive chemicals, severe weathering and abrasion, *plus 33% greater thickness per coat!*

The solids content of 33HB tops all other non-mastic vinyl coatings, yet workable viscosity is retained. It applies smoothly and easily by hot or cold spray. No need to stock two types of materials.

New Lower Cost

In evaluating any coating, always look at the cost per square foot, and the number of coats required to build proper film thickness. 33HB is nearly 24% cheaper per mil square foot than traditionally low cost 33...and compared to other leading vinyls, (see chart) savings are even greater. For example, although the gallon price of Coating "C" is 46% lower than 33HB, because of its low solids content the actual *material* cost for 5 mils thickness is almost 30% higher than 33HB, and the applied cost, (labor and material) is almost 90% higher.

For the full story, send coupon for data on 33HB and detailed cost comparison of the four coatings on the chart.

Amercoat® CORPORATION

4809 Firestone Blvd. · South Gate, Calif.

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Please send me full information on Amercoat 33HB

Name_____

Title_____

Company_____

Address_____

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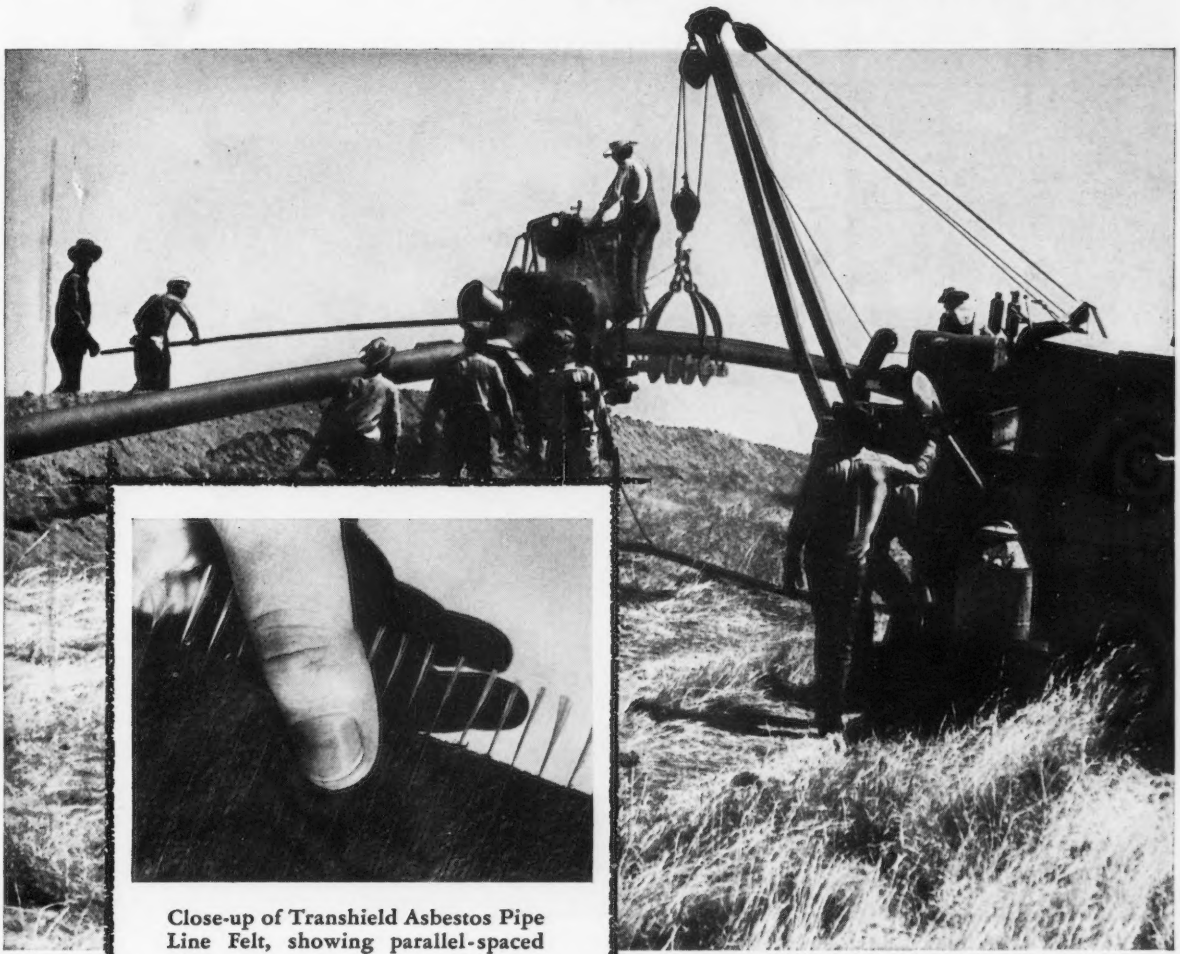
For effective, long-term protection against corrosion . . .



Close-up of Transhield Asbestos Pipe Line Felt, showing parallel-spaced glass yarns.

Coating and wrapping pipe line to check corrosion and assure long-term service.

. . . use Johns-Manville TRANSHIELD® ASBESTOS PIPE LINE FELT

Johns-Manville Transhield is an economical pipe line felt assuring effective, long-term protection against corrosion under average soil conditions.

Designed for easy, high-speed application by modern machine methods, Transhield is strong and highly tear-resistant. Its light weight permits its use in 800-ft. rolls. This advantage cuts roll changes in half for field wrapping equipment.

Transhield provides a protective membrane of coal-tar saturated asbestos felt, reinforced with continuous glass yarns parallel-spaced on ¼" centers. It cannot rot or decay, thus acts as an enduring barrier to shield pipe line enamels from earth loads and soil stress.

For severe soil conditions use Johns-Manville #15 Asbestos Pipe Line felt . . . proven in service for over 30 years.

For further information about Transhield, write to Johns-Manville, Box 14, New York 16, N. Y. In Canada, 565 Lakeshore Road East, Port Credit, Ont.

JOHNS-MANVILLE JM PRODUCTS **Johns-Manville** PRODUCTS FOR PIPE LINE PROTECTION

"These Galvomag anodes are a small investment to protect $20,000 worth of well casings"

Cathodic protection with Galvomag® magnesium anodes is a smart move for any oil well operator.

Corrosion can ruin well casings in a surprisingly short time. Frequently it causes leakage in the casing that floods the producing horizon. Sometimes it causes the casing to collapse. All too often, corrosion results in a costly repair job or abandonment of the well.

A relatively small investment in Galvomag anodes will insure any underground metal structure against corrosion. Galvomag is the name of the Dow high-potential anodes that deliver 25% more current than conventional anodes. In normal soils they give good protection with fewer units. In high-restivity soils they provide the extra current needed to assure good protection.

Contact one of the Dow magnesium anode distributors below for further information and technical assistance, or write to us. THE DOW CHEMICAL COMPANY, Midland, Michigan, Department MA 1427GG.

CALL THE DISTRIBUTOR NEAREST YOU: Cathodic Protection Service, Houston, Texas • Corrosion Services, Inc., Tulsa, Oklahoma • Electro Rust-Proofing Corp. (Service Division), Belleville, N. J. • Ets-Hokin & Galvan, San Francisco, Calif. • Royston Laboratories, Inc., Blawnox, Penna. • Stuart Steel Protection Corp., Plainfield, N. J. • The Vanode Co., Pasadena, Calif.

YOU CAN DEPEND ON DOW

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Corrosion

devoted entirely to corrosion research and control

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Properties and Performance of Polyester Plastics in the Chemical Industry by R. E. Gackenbach and D. C. Estey.

Coal Tar Coatings III—Resistance to Fouling and Degradation by Marine Organisms by R. B. Teel and W. F. Fair, Jr.

Formation of Oxide Films on Chromium and 18Cr-8 Ni Steels by H. J. Yearian, W. D. Derbyshire and J. F. Radavich.

Application of Cable in Cathodic Protection by R. G. Fisher and M. A. Riordan.

How to Determine a "Comparable Cost" for Paints by V. B. Volkening and J. T. Wilson, Jr.

Failure of Steel-Admiralty Duplex Condenser Tubes by Hydrogen Penetration by H. E. Honkala and E. A. Wright.

Cathodic Protection of Oil Well Casings at Kettleman Hills, Cal. by J. K. Ballou and F. W. Shremp.

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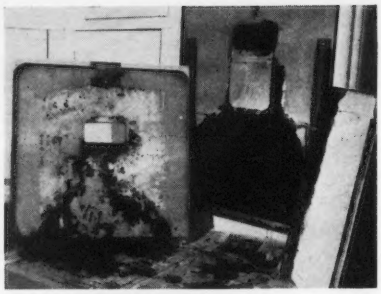

THIS MONTH'S COVER—Rust damaged door of a popular domestic dishwasher. Left: Carbon steel outer shell, thinly painted inside is perforated near vent opening (upper center) and at bottom where insulation formed a poultice soaked with detergent-laden hot water. Stainless inner shell (center) is undamaged. Bottom closure (right) is badly rusted from water dripping through door.

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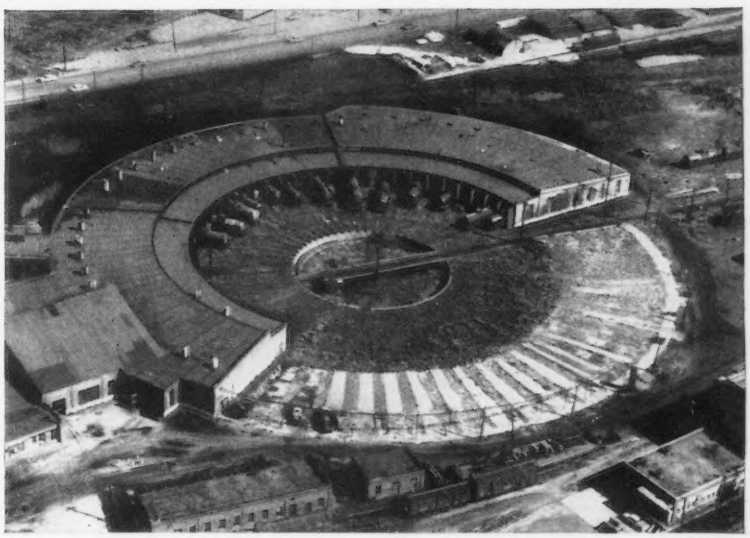
UNICHROME Trade Mark

News about COATINGS for METALS

Metallic.....Organic.....Decorative.....Protective

Phenolic linings improved for tank use

Unichrome Series B-124 Linings give reliable protection — are now being applied by specialists



Ingenuity! Lithcote Corp., a prominent applicator of Unichrome Phenolic Coatings, has adapted a former railroad roundhouse to line tank cars. Cars are sprayed, switched to the baking facilities, and can then be sent rolling back to the customer in short order.

Phenolic-type coatings, the acknowledged "workhorse" for tank lining applications, have a long and successful record in corrosive service. They'll be specified even more now that two problems have been overcome by Metal & Thermit.

(1) Unichrome Phenolic Coatings have been developed that assure more uniform linings, greater impact resistance, and improved protection. (2) Experienced, independent applicators across the country now make it possible to ship large equipment to the nearest specialist, get fast service, save on transportation.

UNIQUE PHENOLIC COATINGS

Several pigmented materials in the Unichrome B-124 Coatings group give 2-mil thicknesses per dry coat. This is twice that of ordinary phenolic coatings. Fewer coats are needed. Clear coatings in the group bake into a hard, smooth film for an easy-to-clean top coat.

BUILT-IN CONTROL

Unichrome Coating B-124-17 is a widely specified phenolic formulation that is gray when wet, olive drab when fully cured. This cure-control offers reliable visual inspection of a job well done. It is valuable for avoiding undercuring in large tanks and tank cars due to uneven heat application.

Details on these Unichrome Linings are given in Bulletin Chem-C-2. Contact Metal & Thermit also for names of nearest applicators of Unichrome Series B-124 Linings.

Reports show Unichrome Plastisols give record service

Unichrome Plastisols, which produce thick vinyl coatings, are withstanding conditions that rapidly deteriorate unprotected metals or other materials. To illustrate:

Plastisol coated ducts exhausting strong acid fumes show no deterioration after 4 years.

After 6 months service, plastisol coated agitators for chemical mixers show absolutely no wear. Rubber coatings, used previously, started to fail after only one month.

Plastisol coating on a 5-ft. bubble tray at bottom of a 60-ft. scrubbing tower has been withstanding acid fumes and caustic solutions for over 2 years now.

A report shows Unichrome Plastisol Coating on racks withstood 6 years of daily use in corrosive plating and cleaning solutions.

APPLICATION NO PROBLEM

Unichrome "Super 5300" Coating spray-applies seamless films as thick as sheet linings. It delivers up to 60 mils per coat. "Series 4000" Plastisols apply by dipping, slushing, troweling or other convenient means. Send for Bulletin VP-2 or name of the nearest applicator of Unichrome Plastisols.

Unichrome is a trademark of Metal & Thermit Corp.

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T-2D-2 Inaccessible Locations, Such as Water Crossings
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T-2D-3 Parallel Lines
L. A. Hugo, Chairman; Phillips Petroleum Co., Bartlesville, Okla.

T-2D-5 Pipe Observed to Be in a Stray Current Field
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T-2E Internal Corrosion of Product Pipe Lines and Tanks
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T-2F Internal Corrosion of Crude Oil Pipe Lines and Tanks
(Election Being Held)

T-2G Coal Tar Coatings for Underground Use
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T-2H-1 Methods to Classify Environmental Conditions
(See chairman T-2H)

T-2H-2 Criteria for Reporting on and Evaluating Underground Coatings
(See chairman T-2H)

T-2H-3 Specifications
(See chairman T-2H)

T-2H-4 Internal Pipe Treatment Specifications
F. B. Burns, Chairman; Kerr-McGee Oil Industries, Inc., Box 125, Wynnewood, Okla.

T-2J Wrappers for Underground Pipe Line Coating
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O. W. Wade, Vice Chairman; Transcontinental Gas Pipe Line Corp., Box 296, Houston, Texas

T-2J-1 Asbestos Felt
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T-2J-2 Glass Wrap
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T-2J-3 Glass Base Outer Wrap
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T-2J-4 Rag Felt
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T-2J-5 Rock Shield
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T 3 General

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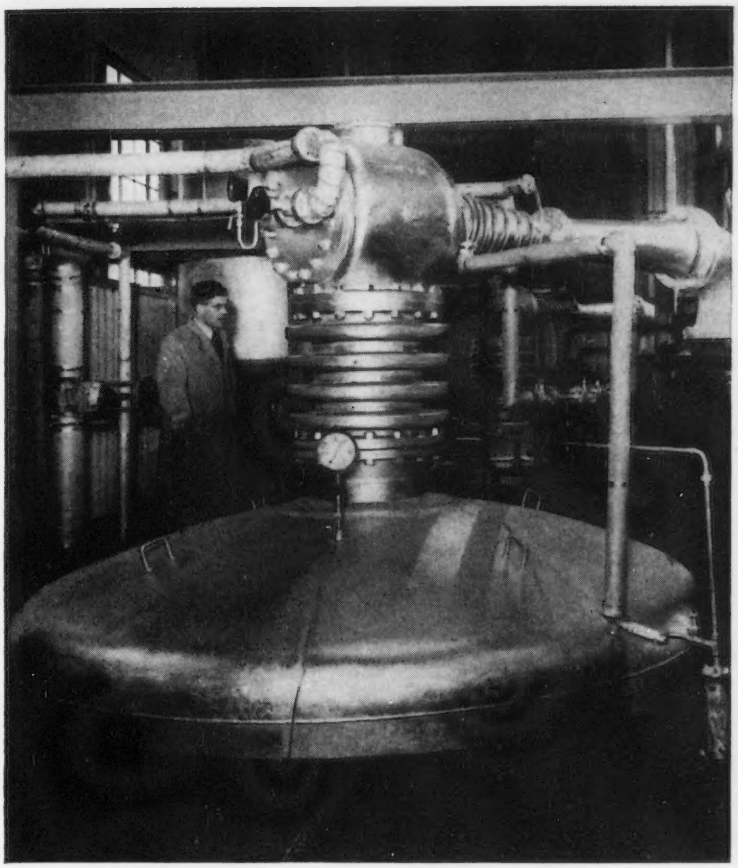
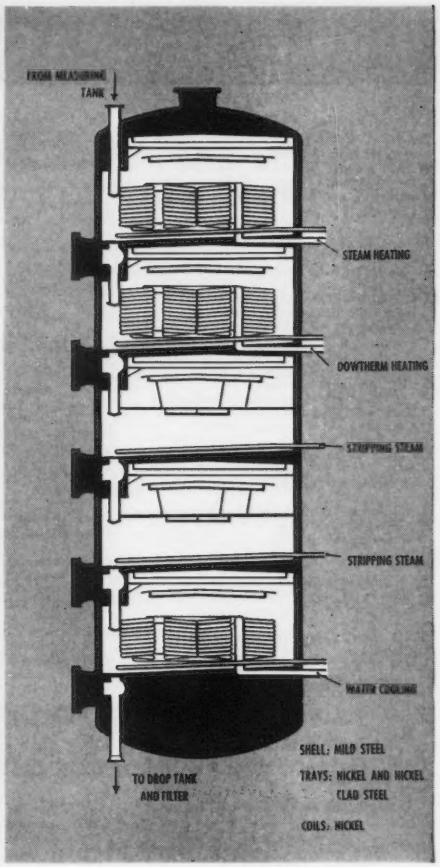
(Continued on Page 8)





Nickel protects purity of vegetable oils during deodorization in these Southern Oils, Ltd. stills. Diagram shows general arrangement and where nickel is used in the units. Fabricator: Rose, Downs & Thompson, Ltd., Hull, England. Design: Girdler Corporation of America.

Processed in nickel, foods and other organics stay fresh longer

One of the most baffling problems in converting and purifying vegetable oils for human consumption is presented by the equipment itself.

Even minor corrosion of this equipment may build up metallic concentrations in the product sufficient to create new threats to purity. High process temperatures amplify the danger.

The harm shows up in storage. Early rancidity. Undesirable color changes.

Processing in nickel equipment provides double protection

Nickel acts in two ways to prevent trouble of this kind.

1. Nickel resists corrosion by fatty acids and other corrosives in vegetable oils and many foods . . . even at elevated process temperatures. With nickel, metallic concentrations usually stay far *below* the harmful level.
2. Nickel doesn't promote rancidity and color changes. It's non-toxic, too.

Southern Oils, Ltd. (Manchester, England) is one of many companies that takes advantage of these properties to insure high product purity. In their vegetable oil deodorizing stills, for example, (see photo and drawing) tray sides are nickel-clad steel plate, tray base and heating coils pure nickel. So are many of the auxiliary fittings . . . tray tops, liquid level probes, etc.

Despite process temperatures that run as high as 475-480°F., Southern Oils' storage experience with the product has been excellent.

To learn what many food processors are doing to solve their corrosion and purity problems, send for a copy of Inco's Technical Bulletin T-28. It's yours for the asking. And if you have a specific question, why not jot it down? We'll do our best to help you.

The International Nickel Company, Inc.
67 Wall Street New York 5, N. Y.

INCO TRADE MARK **Nickel**—for purity

Directory of NACE Technical Committee Officers

(Continued From Page 6)

T-3A-1 General Theory of the Action of Corrosion Inhibitors
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T-3A-2 Methods of Testing & Screening Corrosion Inhibitors
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T-3A-3 Materials Available for and Application of Corrosion Inhibitors
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T-3F-3 Corrosion Products
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T-3F-6 Intercommittee Activities
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T-4A-1 Recommendations for Materials to Be Used for Electrical Grounding to Reduce Corrosion
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T-4A-2 Recommendations for Grounding on Private Premises
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T-5B-5 Corrosion by Molten Salts and Metals
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T-5C Corrosion by Cooling Waters

T-5C-1 Corrosion by Cooling Waters (South Central Region)
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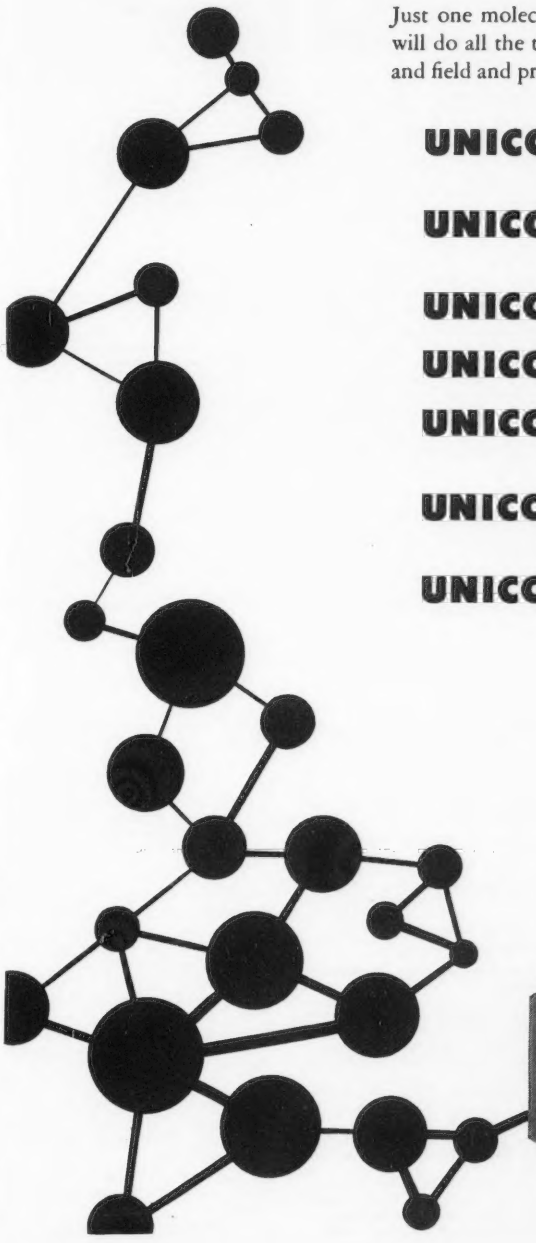

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(Continued From Page 8)

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T-6F-4 Safety

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T-6G Surface Preparation for Organic Coatings

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T-6H Glass Linings and Vitreous Enamels

T-6J Protective Coating Application Problems

T-6J Los Angeles Area, Protective Coating Application Problems

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T-6R Protective Coatings Research

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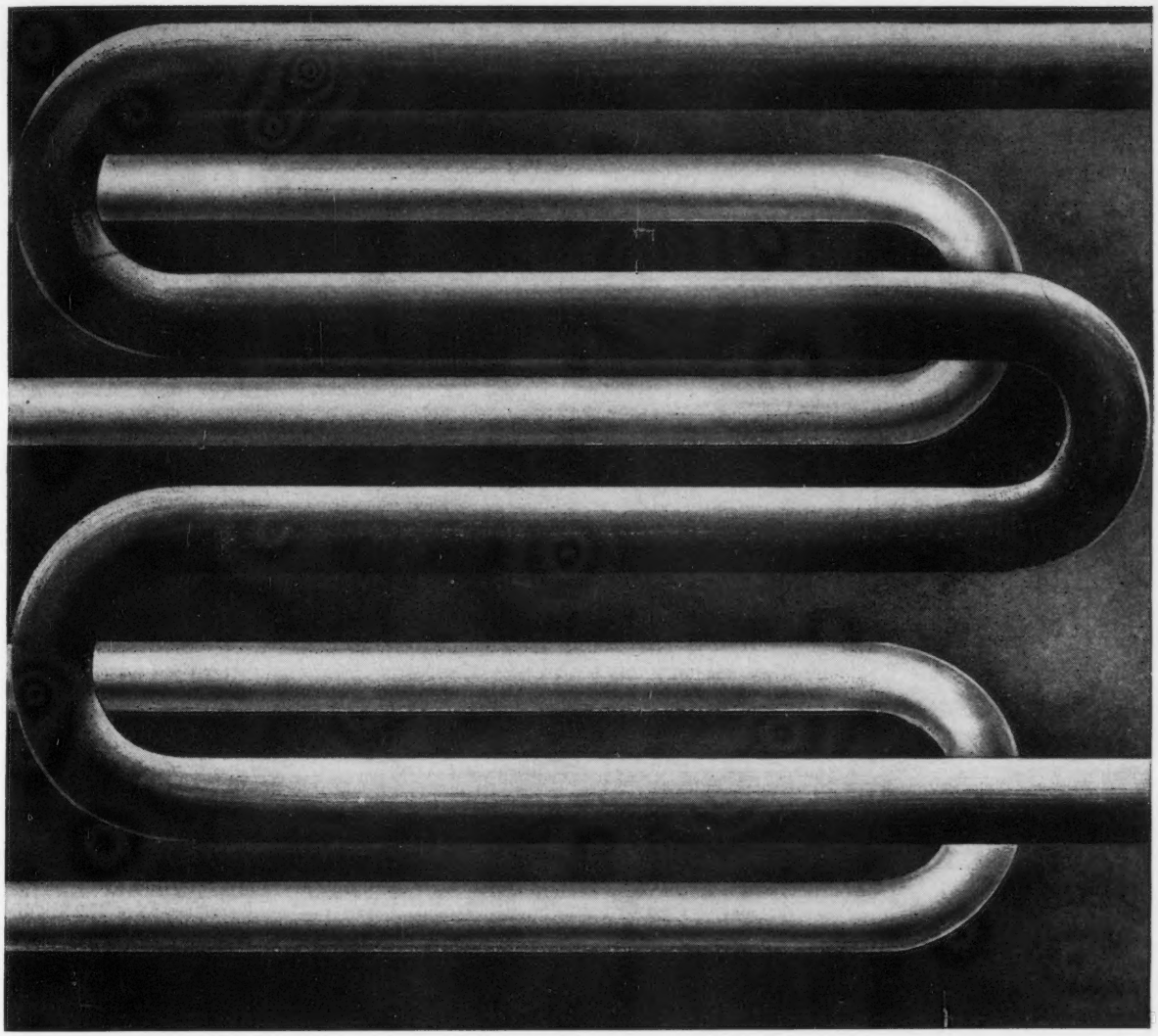
(Continued on Page 12)



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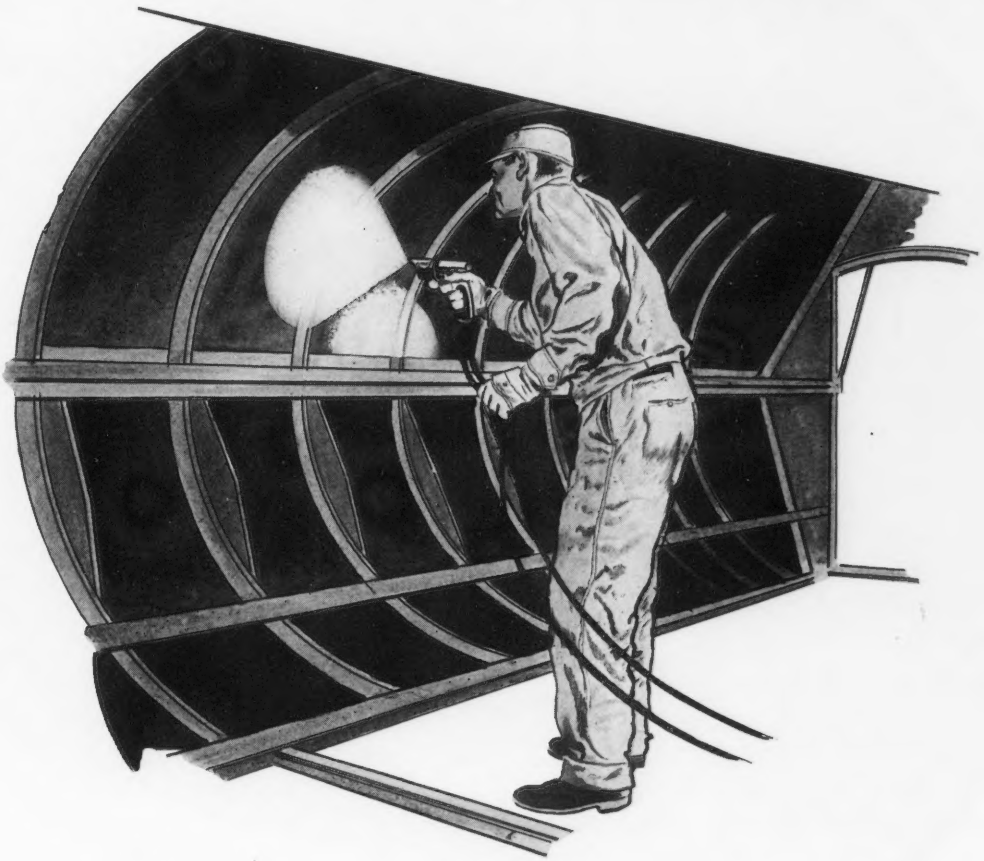
Directories of NACE Regional and Sectional Officers are published in CORROSION on alternate months beginning with February each year. Directories of Technical Committee Officers are published on alternate months beginning with January. On the months when the technical committee officers' names appear an effort is made to include also (as above) names of regional and sectional officers that have changed since last publication so those interested may know about changes. Names of NACE national officers and board members are published monthly on the page facing the first page of the Technical Section.



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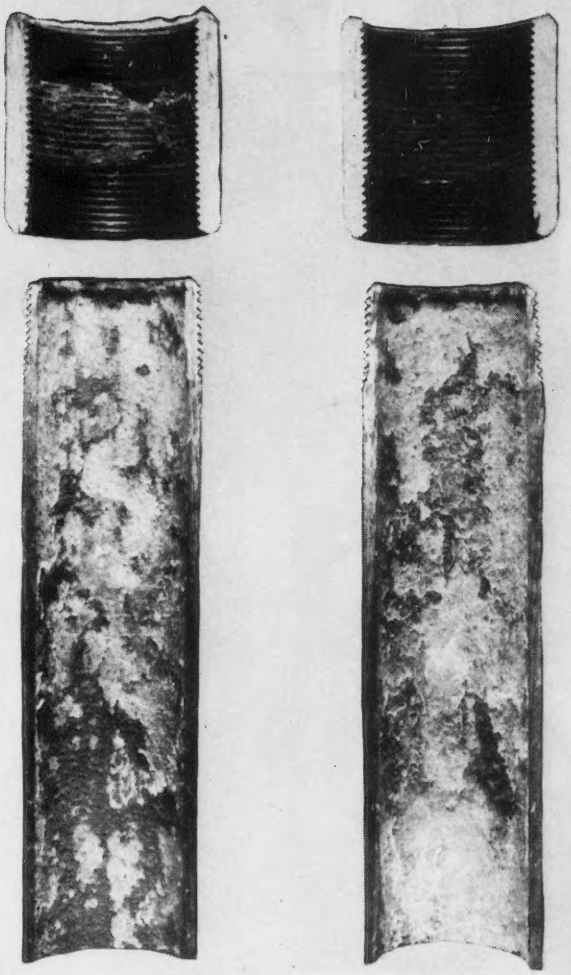
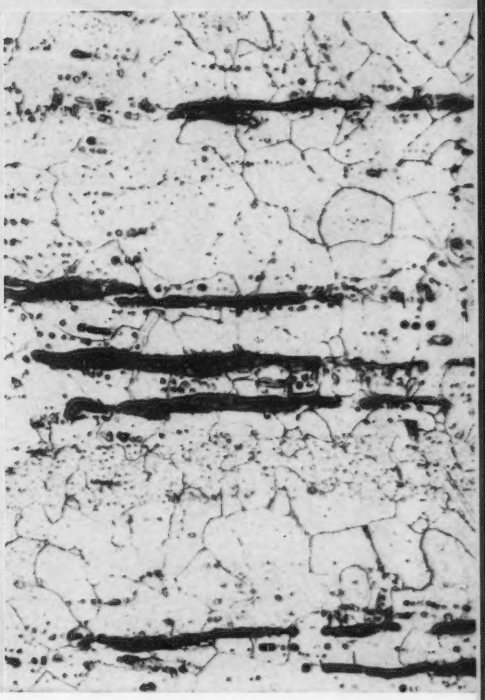
another Wrought Iron case against corrosion



30 YEARS OF SERVICE

Longitudinal photomicrograph U-44-2 (100x) shows a typical area—parallel to rolling — of the microstructure observed in the subject 1½" wrought iron pipe.



Metallurgical Report 6900 – Hot Water Line*

This report deals with the metallurgical examination of a length of 1½" wrought iron pipe with coupling, submitted to our laboratory for investigation and comment.

The sample submitted had been in service for 30 years as a hot water line, yet was found to be in excellent condition. The inner surface of the pipe showed light, tightly adherent products of corrosion. Underlying galvanizing on the inside of the pipe was virtually intact.

Drillings taken from the pipe analyzed as follows: Carbon—.018%; Manganese—.050%; Phosphorus—.142%; Sulphur—.018%; Silicon—.174%; Iron Silicate—3.45%.

Wrought iron pipe's 30 years of unfailing service in this installation may be traced to its unique defensive network of glasslike iron silicate fibers. Service records such as this one present a very strong case for wrought iron pipe's outstanding corrosion resistance. A. M. Byers Company, Clark Building, Pittsburgh 22, Pennsylvania.

*Name of actual installation given on request.

Write for free cloth-bound book, *Wrought Iron: Its Manufacture, Characteristics and Applications.*

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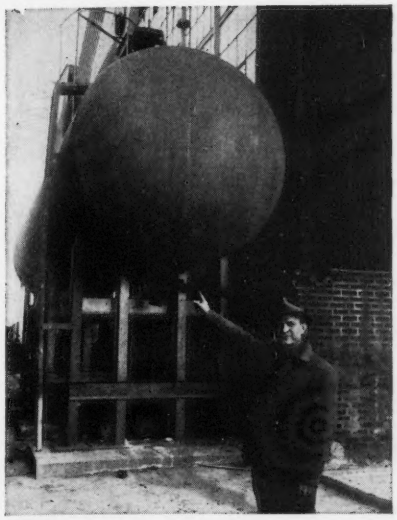
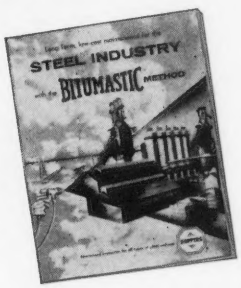


Acid Storage Tank at pickling plant was given heavy-duty protection against corrosion and spillage by a coating system of Bitumastic® No. 50. This coal tar base coating gives exceptional dry-film thickness with each coat, its 20-25 mil protective layer exceeding that obtained with ordinary paints by as much as 8 times. For this reason, Bitumastic No. 50 is used where unusually severe corrosive conditions require the maximum protection available in an economical, cold-applied coating system.



Corrosion Control in metalworking plants, with particular emphasis on the problems of the basic metals producing industry, is the subject of a new booklet now available from Koppers Tar Products Division. It outlines typical specifications for the protection of various surfaces and reviews application techniques to insure long-term protection of plant structures and equipment. For your copy, use coupon below.

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Please send me "The Bitumastic Method to Prevent Corrosion in the Steel Industry."

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how to CUT CORROSION LOSSES

a report by Koppers Company, Inc.

Number 11 of a series

H. J. Cibula
Sales Representative

One of the toughest problems to face the corrosion engineer is protection of steel pickling plants. The dense vapors created by the hot sulfuric acid used can deteriorate the heaviest structural member quickly. Here is a case history of how one company solved this corrosion problem.

* * *

One of the nation's oldest and largest pickling and hot-dip galvanizing firms made a careful study of the problem with the aid of its consulting engineering firm. Past experience has been that little more than eleven years of useful service could be expected from structural members in pickling sheds: the study was undertaken to determine the best protective coating system for new facilities planned.

The first step was to test a wide variety of coating materials under actual service conditions in one of the company's existing installations. The systems tested included expensive multi-coat types, lead-base, synthetics, and coal-tar base coatings. Not only were coatings tested, but various surface preparation and application techniques were compared for economy and effectiveness. Ease of application was of considerable importance since the firm planned to have its own maintenance personnel do the coating job.

On review of test results, both company and engineering consultant personnel selected Bitumastic® Super Service Black, a coal-tar base coating, for its economy, performance, and ease of application.

* * *

Structural members delivered to the new building site were *completely* cleaned by pickling and neutralizing in the firm's own plant: the importance of thorough surface preparation has also been proven in the test. One coat of Bitumastic Super Service Black was applied by spraying *before* the steel was erected. (Note that the priming step is eliminated with Bitumastic coatings.)

Coating the steel before erection was fast and economical since there was no interference with connecting members. After erection, second and third coatings of Bitumastic Super Service Black were applied to obtain desired system thickness.

* * *

This case history points up the fact that many plant areas previously considered "expendable" can be economically and effectively protected against corrosion. Today, with the high cost of materials and labor, the savings that can be realized by such engineered corrosion-prevention techniques are significant.

If you have a particular corrosion control problem, write: Koppers Company, Inc., Tar Products Division, 1450 Koppers Building, Dept. 100G, Pittsburgh 19, Pa. District Offices: Boston, Chicago, Los Angeles, New York, Pittsburgh and Woodward, Ala. In Canada: Koppers Products, Ltd., Toronto, Ontario, and Edmonton, Alberta.

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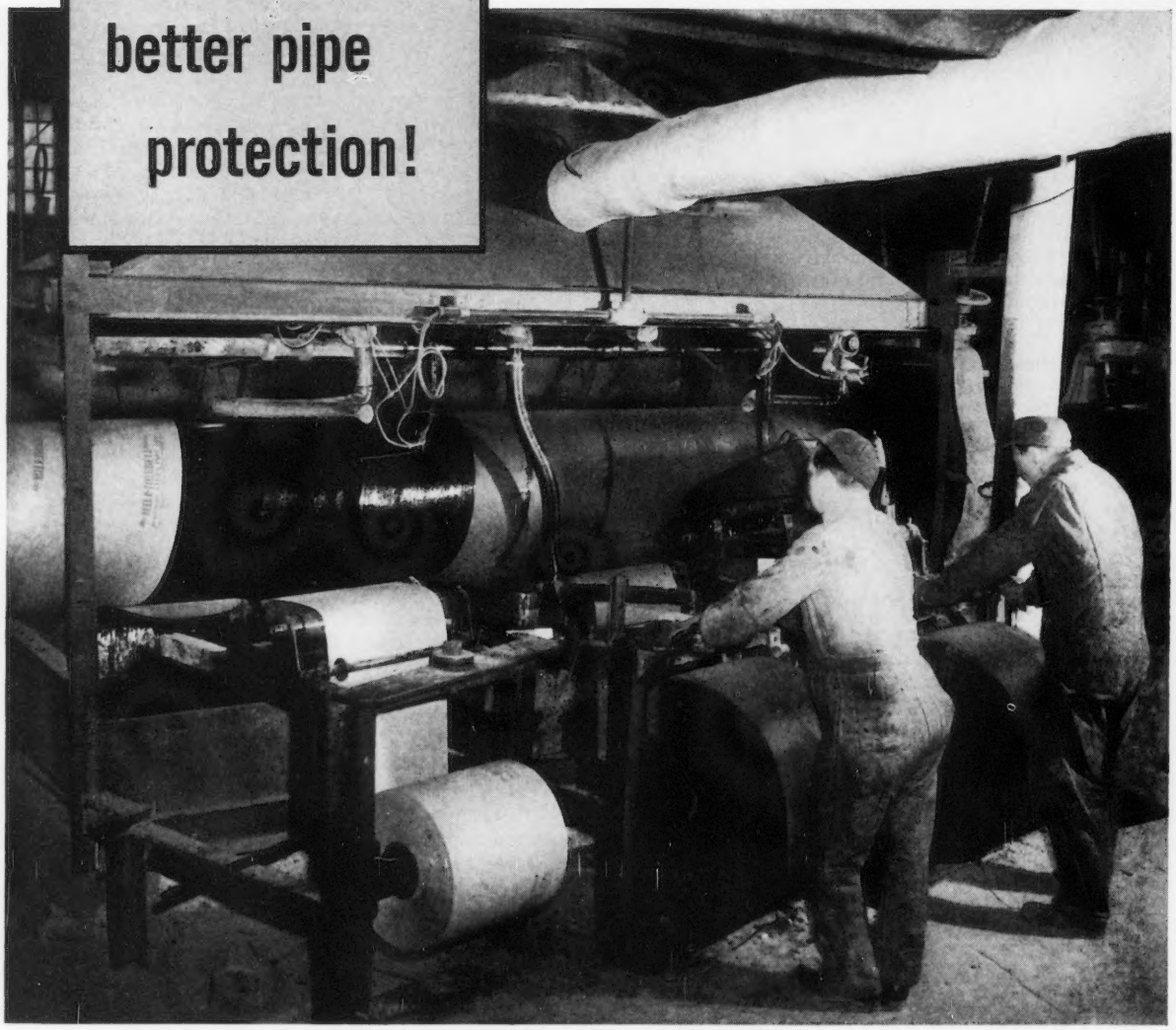
This is another first by Hill, Hubbell, another reason to specify Hill, Hubbell wrapped pipe.



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THE NATIONAL ASSOCIATION OF CORROSION ENGINEERS

is a non-profit, scientific and research association of individuals and companies concerned with corrosion or interested in it, whose objects are:

(a) To promote the prevention of corrosion, thereby curtailing economic waste and conserving natural resources.

(a) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.

(c) To encourage special study and research to determine the fundamental causes of corrosion, and to develop new or improved techniques for its prevention.

(d) To correlate study and research on corrosion problems among technical associations to reduce duplication and increase efficiency.

(e) To promote standardization of terminology, techniques, equipment and design in corrosion control.

(f) To contribute to industrial and public safety by promoting the prevention of corrosion as a cause of accidents.

(g) To foster cooperation between individual operators of metallic plant and structures in the joint solution of common corrosion problems.

(h) To invite a wide diversity of memberships, thereby insuring reciprocal benefits between industries and governmental groups as well as between individuals and corporations.

It is an incorporated association without capital stock, chartered under the laws of Texas. Its affairs are governed by a Board of Directors, elected by the general membership. Officers and elected directors are nominated by a nominating committee in accordance with the articles of organization. Election is by the membership.

Inquiries regarding membership, and all general correspondence should be directed to the Executive Secretary at the administrative headquarters of the National Association of Corrosion Engineers at 1061 M & M Building, No. 1 Main Street, Houston 2, Texas.

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Corrosion

OFFICIAL MONTHLY PUBLICATION

National Association of Corrosion Engineers

An Association of Individuals and Organizations Concerned with Control of Corrosion

1061 M & M Building Houston 2, Texas

Vol. 12 July, 1957 No. 7

Topic of the Month

Corrosion Experience With a Mixing Nozzle For Dilute Sulfuric Acid

By W. E. GIBSON*

THE MANUFACTURE of synthetic rubber is accomplished by copolymerizing a mixture of butadiene and styrene in a soap solution containing certain other chemicals under controlled conditions. After the copolymerization cycle is completed the unreacted butadiene and styrene are removed, leaving an emulsion called "latex." This is a viscous liquid containing a high percentage of solids.

In order to produce the finished, or dry rubber, a stabilizer is added to the latex, together with other constituents depending on the type of rubber desired. The latex is then coagulated by a dilute solution of brine and sulfuric acid, forming crumb rubber, which is then washed, filtered, dried and compressed into bales as the final product.

The coagulant is maintained at the proper acid concentration (0.05 percent) and temperature (120 F) in a 316 stainless steel lined dilute acid make-up tank. Figure 1 is a simplified diagram showing the method used. Ninety-five percent sulfuric acid is fed into the liquid as required through a dip pipe or mixing nozzle.

A series of tests were conducted to alleviate or eliminate the corrosion associated with the handling of the dilute sulfuric acid solution. Although some corrosion takes place in the stainless steel lined tank, the rate is such that payout for a more inert material is not justified. With the exception of the dip pipe, the corrosion has been controlled in most of the equipment by the application of various Epon (R) resin coatings.

Numerous materials were investigated for possible use in fabricating the dip pipe. Available corrosion data gave the corrosion rate of 316 stainless steel as

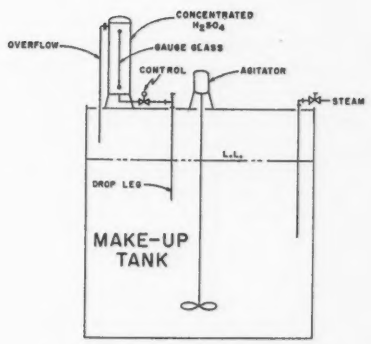

Figure 1—Diagram of a 316 stainless steel lined make-up tank.

Figure 2—Corrosion of a 316 stainless steel dip pipe after two months' service.

* Shell Chemical Corporation, Torrance, California.

Figure 3—Corrosion of an Hastelloy D dip pipe after three months' service.

Figure 4—Steel pipe electrostatically coated with a vinyl plastic.

Figure 5—Corrosion of pipe pictured in Figure 4 after 36 days' service.

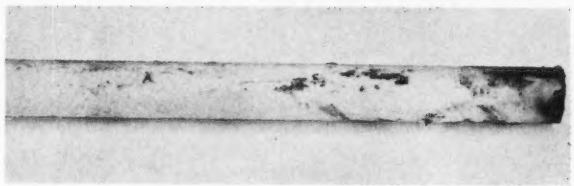

Figure 6—Teflon dip pipe after several months' service.

0.02 inch per year. Therefore this material appeared to be acceptable and installation justified. Figure 2 shows the extensive corrosion that had occurred after two months' service. Hastelloy D with an estimated corrosion rate of less than 0.002 inch per year was next placed in service with the results after three months' service shown in Figure 3. Upon the recommendation of a manufacturer a steel pipe was electrostatically coated with a vinyl plastic (Figure 4). As shown in Figure 5 complete failure occurred after 36 days' service.

Since all of the failures occurred at, or near, the tank liquid level, it was assumed that the heat of dilution resulting from the mixing of the 95 percent acid and the dilute solution with insufficient agitation resulted in localized temperature rise sufficient to accelerate corrosion of the dip pipe. Subsequently a recording thermometer was installed on the dip pipe and it was found that, upon introduction of the 95 percent acid, there would occur an average temperature rise up to 260 F.

The answer to the problem discussed above proved to be a dip pipe fabricated from Teflon. This material has been in service since November 1956 and to date shows no indication of corrosion.

Corrosion in Light Oil Storage Tanks*

By E. H. TANDY

Introduction

WITH FEW exceptions, the rate of corrosion on the shells of oil storage tanks handling finished and semi-finished stocks varies directly with the API gravity of the oil for any given location. Tanks handling heavy black oils (10 degrees API and heavier) have shell corrosion rates of less than 1 mpy while gasoline tanks (55 degrees API and lighter) may have rates as high as 20 mpy. The most important factors governing corrosion rates in storage tanks are:

1. Solubility of oxygen in the oil.
2. Rate of working (frequency and rate at which the tank is filled and emptied.)
3. Type of roof.
4. Vapor pressure of the oil.
5. Location of the tank (climatic conditions.)

Corrosion on the wetted portion of tank shells takes place continually and the rate depends on the ability of the oil to supply water and oxygen for the reaction. It has been demonstrated that the oil itself does not enter into the reaction[1] but simply acts as a vehicle for the corrodants. The solubility of water in oil is about the same for all stocks regardless of the API gravity and varies directly with the temperature. The solubility of oxygen on the other hand varies directly as the API gravity and inversely as the temperature (see Tables 1 and 2.)

Generally speaking, oils that are stored at a temperature of 150 F or less are stored over a bottom water layer and are therefore, normally saturated with water for any given temperature. In addition, water is constantly being added to the tanks with each new batch of oil and by condensation from the vapor space. The oil is saturated with water from processing and since it is generally warmer than the normal storage temperature, water is precipitated out of the oil as it cools. This necessitates removing excess water from the tank from time to time.

Oxygen, on the other hand, may be present in the oil in small quantities after processing but it is primarily absorbed into the oil from the vapor space. The oxygen content in the oil will vary depending on the ability of the oil to absorb it, the amount of air in the vapor space and the amount of natural mixing or diffusion in the oil.

Corrosion in run down pans which receive the oil directly from the distillation and treating plants, is also dependent on the water and oxygen content of the oil. However, other factors such as the presence of H_2S, low pH due to residual chemicals, warmer oil temperature, etc., which are not present in the finished stocks have a profound effect on the corrosion rate. For this reason they will not be considered here.

Kerosenes and heavier oils (50 degrees API and heavier) do not present any particular problem due to corrosion. The corrosion rate is low (about 3 mpy or less.) The maximum corrosion usually occurs on the top course and on the roof and supports of cone roof tanks. In floating roof kerosene tanks the corrosion rate approaches that of gasoline in the lower third of the tank and then drops rapidly to the atmospheric rate near the top. The mechanism of corrosion for these tanks is essentially the same as shown for gasoline tanks below.

It is recognized that the following observations are for tanks situated on or near the sea coast. Also, because of air pollution, cone roof tanks are no longer used for light oil storage unless they are equipped with some form of vapor recovery system. While the corrosion rates for these tanks may be somewhat higher than for those located inland, the corrosion mechanism should be the same. It is interesting to note that the corrosion rates reported

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Abstract

The most important factors governing corrosion rates in oil storage tanks are solubility of oxygen in the oil, rate of working, type of roof used, vapor pressure of the oil, and location of the tank (climatic conditions.) Data are given to show the relationship between stocks contained in tanks versus corrosion rate, and size and location of tanks versus corrosion rate. A corrosion mechanism is considered briefly. Corrosion control measures discussed include gas blanketing, inhibitors, breathing systems and protective coatings. Other topics considered include corrosion below the liquid level, vapor space effects, temperature effects and plate thickness corrosion allowance. 4.4.6

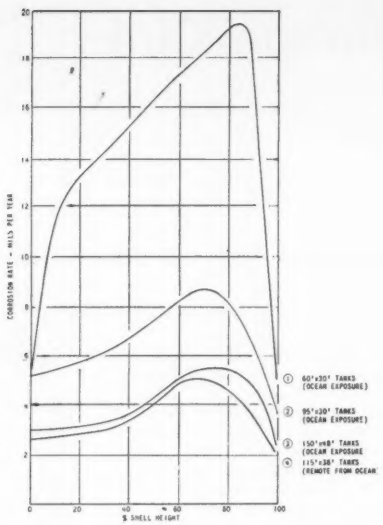
CORROSION RATE - MILS PER YEAR
% SHELL HEIGHT
① 60'×30' TANKS (OCEAN EXPOSURE)
② 95'×30' TANKS (OCEAN EXPOSURE)
③ 150'×48' TANKS (OCEAN EXPOSURE
④ 115'×30' TANKS (REMOTE FROM OCEAN

Figure 1—Corrosion rate for cone roof gasoline tanks.

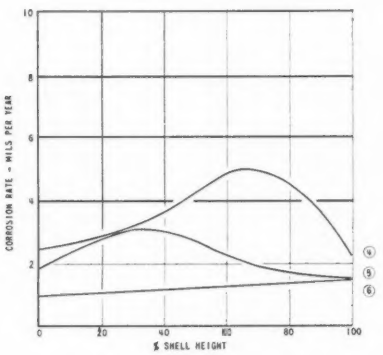

Figure 2—Corrosion rate for gasoline and mixed light oils (remote from the ocean). Code to curves is as follows: Curve No. 4—115 foot by 30 foot cone roof with all gasoline service (from Figure 1); Curve No. 5—115 foot by 30 foot floating roof with 46 percent gasoline, 19 percent kerosene and 35 percent gas oil service; Curve No. 6—120 foot by 40 foot cone roof with 43 percent gasoline, 14 percent kerosene and 43 percent gas oil service.

TABLE 1—Solubility of Water in Gasoline*

Temperature (Degrees F)	cc/liter	Temperature (Degrees F)	cc/liter
40	0.043	80	0.071
50	0.050	90	0.079
60	0.057	100	0.086
70	0.064	110	0.095

* Converted from Wachter & Smith[1].

TABLE 2—Solubility of Oxygen[2] in cc/liter at 77 F

Partial Pressure, (millimeters)	Water (10 API)	Gas Oil (30 API)	Pentane (93 API)
100	6.0	20.3	70.0
160		32.0	123.0
400		81.0	304.0
760		154.0	576.0

here and those reported for gasoline pipe lines[1] and tank ships,[3,4] are in the same order of magnitude.

Corrosion Versus Stocks

Solvent and gasoline tanks (50 degrees API and lighter) have the highest overall shell corrosion rates and create the major maintenance problems. For this reason this paper will be confined mainly to a discussion of such service. Corrosion in these tanks occurs over the full height of the shell but to varying degrees. The corrosion rates vary from 3 mpy to 6 mpy for semi-finished stocks and usually range from 3 mpy to 10 mpy for finished stocks but may run as high as 20 mpy for some types of service.

Generally speaking, the heavier stocks in floating roof tanks will have the lowest corrosion rate. The lighter stocks (55 degrees API and lighter) in small cone roof shipping or blending tanks have the highest corrosion rate. Figure 1 illustrates typical corrosion curves for cone roof gasoline tanks. Figure 2 shows curves for cone roof tanks in gasoline service exclusively (4), and curves for floating roof (5) and cone roof (6) tanks in mixed gasoline and light oil service. These curves represent the *average* corrosion rates for the various groups of tanks and are based on actual measurements of the shell plates after 10 years or more of service. Most of the gage measurements were taken after a service life of 20 years or more.

Corrosion Versus Size and Location

The curves in Figures 1 and 2 illustrate the effect of size, service and location. The tanks which furnished data for Curves (1), (2) and (3) on Figure 1 are all located in the same general area which is on a hill about 150 feet above sea level and about a half mile from the beach. The tanks represented by Curves (4), (5) and (6) in Figure 2 are all about 1½ miles from the beach and about 100 feet above sea level. Curves (1), (2) and (3) indicate the effect of size on the corrosion rate. However, other factors are important as well.

The small tanks represented by Curve (1) are used to blend aviation and other high octane gasolines. Because of their size and service they are filled and emptied quite rapidly thus promoting greater turbulence and breathing. Other small tanks in the same area which work slowly and with slightly heavier stocks have corrosion rates which approach those for the larger tanks in Curve (2). The tanks represented by Curve (3) work at the same rate as those in Curve (2) but handle slightly heavier stocks. The tanks represented by Curve (4) are used to store the base stocks which are blended to make up the finished stocks handled by the other tanks. The fact that they are a considerable distance from the ocean and work more slowly than the others accounts for their lower corrosion rate.

The distance from the beach is important because it is directly related to the amount of salt spray in the air breathed by the tanks. The normal onshore breeze blowing most of the day and evening is gentle (about 5 to 6 mph) and does not propel the salt spray for more than a half mile or so from the surf. Hence,

the tanks near the beach breathe in some salt spray with the air which either drops directly into the oil or condenses out on the exposed surfaces. In either case it acts as a corrosion accelerator.

Typical Tank Scales

The shells of cone roof gasoline and solvent tanks are covered with a continuous layer of scale which has the same general appearance and texture regardless of the type of service. In the lower part of the tanks (see Figure 3) tubercules are present which contain ferrous hydroxide ($Fe(OH)_2$) adjacent to the metal under a hard cover of hydrated ferrous and ferric oxides. Elsewhere (see Figure 4), the scale will consist of a thin (0.02 inch to 0.06 inch) hard, tightly adhering layer of black scale which has been identified as magnetite ($Fe_3O_4 \cdot n \cdot H_2O$). Over this is a layer of red-brown scale which varies in texture from a rather porous material on the metal side to a hard dense material on the stock side. This layer may be as much as ⅜ inch to ½ inch thick and has been identified as mostly hematitie (Fe_2O_3). The thickness of the scale depends on the length of time the tank has been in service and the amount of corrosion that has taken place. The underlying metal is generally roughened and pitted (see Figure 5).

Corrosion Mechanism

Corrosion in gasoline tanks is very similar to that found in gasoline pipe lines. It has been demonstrated that gasoline takes no part chemically in the reaction.[1] Water and oxygen must be present for the corrosion reaction to occur at all. The corrosion reactions can be expressed as follows:

$$2Fe + 2H_2O + O_2 \rightarrow 2Fe^{++} + 4OH^- \rightarrow 2Fe(OH)_2 \rightarrow 2FeO \cdot H_2O \quad (1)$$

$$3FeO + H_2O \rightarrow Fe_3O_4 + H_2 \quad (2)$$

$$2Fe_3O_4 + O_2 + H_2 \rightarrow 3Fe_2O_3 \cdot nH_2O \quad (3)$$

The formation of magnetite in the scale is important to the reaction because it is cathodic with respect to the steel shell and has been found to accelerate the corrosion reaction.[5]

Below Liquid Level

In looking at the corrosion curves for cone roof tanks it can be seen that the corrosion rate near the bottom of the tank is low but still higher than the atmospheric corrosion rate (2 to 4 mpy). In this area, corrosion occurs continually at a slow but fairly constant rate. A constant supply of water is available from the bottom water layer but only a limited amount of oxygen. This is because this area on the average is at a considerable distance from the vapor space which is the source of supply of oxygen. The character of the scale, which is predominately hydrated ferrous and ferric oxides, is indicative of these conditions. Further up on the shell where the corrosion rate approaches a maximum, the scale is composed mainly of ferric oxides. Since this area is close to the surface of the oil it receives a larger supply of oxygen and can be expected to corrode more rapidly while it is submerged. But, as shown on Figure 6, this area is submerged only about 20 percent of the time and therefore, the corrosion rate would have to be about 10 times faster while submerged than the corrosion rate on the first course. This does not appear reasonable. The corrosion rate in this area then must be higher than the bottom course rate while submerged and corrosion must continue at some rate well above the atmospheric rate (2 to 4 mpy) while it is exposed.

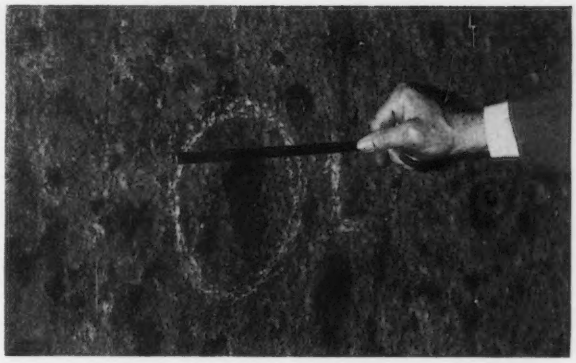

Figure 3—Sixty foot by 30 foot cone roof tank in gasoline mixing service for seven years. Photo shows thick rough scale with tubercules on the bottom course. Total loss was 0.09 inch; corrosion rate was 13 mpy.

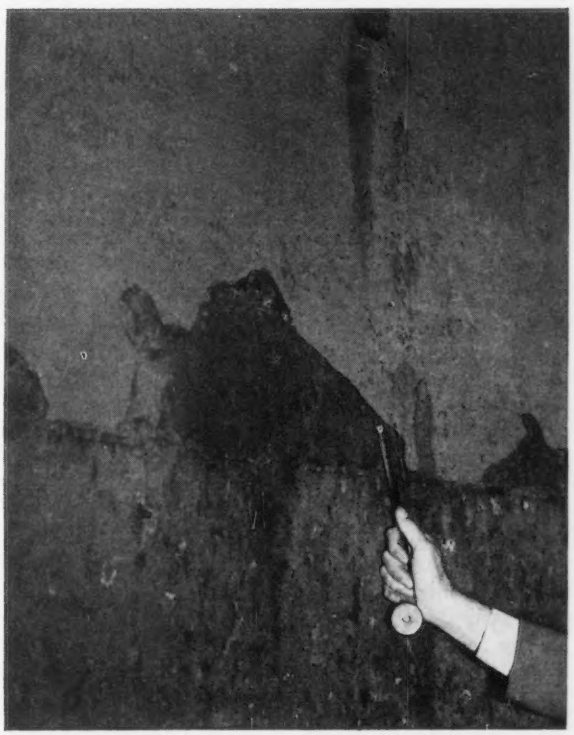

Figure 4—Same tank as shown in Figure 3. Note thick hard scale which was about ⅜-inch thick on the third course. Shell had lost 0.14 inch; corrosion rate was 20 mpy.

Vapor Space

The underside of the roof and the totally exposed portion of the shell have very little scale and the corrosion rate (2 to 5 mpy) is about the same as the

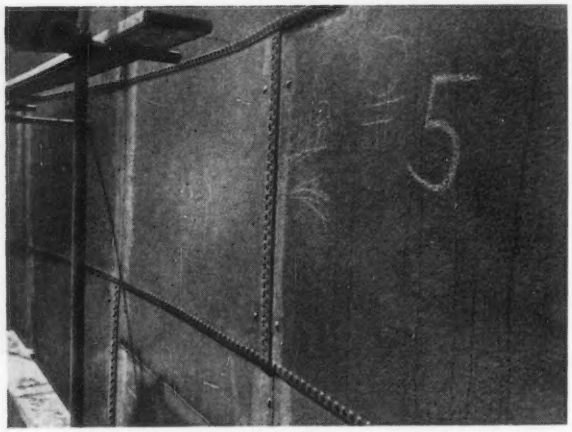

Figure 5—One hundred twenty foot by 41 foot tank in mixed gasoline service for 31 years. Photo shows generally roughened and pitted surface after sandblasting. Total shell loss was 0.14 inch; corrosion rate was 4 mpy.

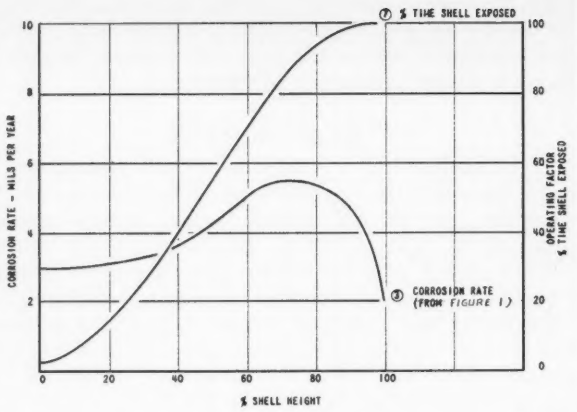

Figure 6—Relation between the time shell is exposed and corrosion rate for cone roof tanks.

atmospheric rate. Below the top liquid level of the tank and progressing downward, the amount of scale on the shell increases rapidly up to a maximum at the point of maximum corrosion (about 60 percent to 80 percent of the shell height). It appears then, that while the partly submerged corrosion rate is higher than for the totally submerged part of the shell, there is also appreciable corrosion while the shell is exposed.

The daily breathing cycle of a gasoline tank as explained by Prater and Huff[6] causes large temperature fluctuations in the vapor space and the consequent evaporation and condensation of the gasoline. The composition of the vapor is about half air and half gasoline vapor on the average. The temperature of the vapor space, which is important in the corrosion reaction,[7] varies with the ambient temperature and may be as much as 20 degrees F above the ambient at the hottest part of the day.

During the night when the temperature of the vapor-air mixture drops below the dew point, gasoline saturated with air condenses on the under side of the roof and on the exposed portions of the shell. Since the corrosion rate on the under side of the roof is about the same as the atmospheric rate, the condensed gasoline does not contain sufficient moisture for an appreciable corrosion reaction. Nor does it stay wet long enough. At the point of maximum corrosion the shell is exposed about 80 percent of the time and while exposed must have the same amount of condensation as the roof. But, since it has a much higher corrosion rate, there must be some additional factor causing the corrosion. The obvious answer is the scale. While the shell is covered with oil—and this may be for days or weeks at a time—a layer of corrosion product is built up. When the liquid level drops and the scale is exposed, oxygen is absorbed from the vapor space thus converting the hydrates to oxides and the ferrous oxides to ferric.

It is reasonable to believe that as the layer of scale increases in thickness the amount of oxygen that can diffuse into it directly from the vapor space becomes limited thus slowing the reaction appreciably. However, gasoline which condenses on the shell and contains appreciable amounts of oxygen can easily penetrate the scale and carry the oxygen with it for the reaction. While the shell is exposed, the sun or the atmospheric temperature may evaporate the gasoline and the increased temperature will drive the oxygen out of solution inside the scale where it can react. This is probably almost a daily cycle. Each night more oxygen loaded gasoline condenses on and penetrates the scale and each day it is driven out. Naturally, equilibrium is reached when all the water in the scale is reacted and all the ferrous scale is converted to ferric. But since the scale is alternately submerged and exposed by the normal working cycle of the tank, it seems reasonable therefore, that the corrosion process is continuous but with varying degrees of activity.

Temperature Effects

The importance of condensation on the upper courses of tanks is illustrated by the fact that the highest corrosion rate in the upper courses is usually found on the north side. In several instances the lower part of the top course of a tank has been found perforated while the south side of the same course had lost only about half of its thickness. In all cases the perforations have been below the top liquid level of the tank. Also, the roof and the unwetted portion of the top course had not had appreciable corrosion.

Corrosion during the submerged periods is also a continuous process. Because of the daily temperature cycle, natural convection currents are set up[6] in the gasoline which carry water up from the bottom and oxygen down from the surface of the oil. Oxygen is absorbed directly into the oil from the vapor space and is brought in by the condensed gasoline dropping from the vapor space as well. During the day the shell warms, thus warming the oil layer adjacent to it and causes it to rise. This action also causes the oil to lose oxygen because of the increased temperature. The oxygen is dropped out against the shell since that is the hottest part.

During the night the shell cools thus chilling the

oil and causes it to sink. Because of the decreased temperature, water is dropped out and since the shell is now the coldest part, it is dropped out on or adjacent to it. Once the scale layer is built up the evolution of oxygen and the precipitation of water will take place mostly in the scale which acts as a reservoir. It is probable also that scale acts as an insulator, thus accentuating the temperature difference between the shell and the oil in contact with it. New supplies of both water and oxygen are constantly being added by the natural convection currents of the oil, thus promoting a continuous corrosion reaction. The speed of the reaction at any point and at any time will vary depending on the temperature of the scale and the concentration of the water and oxygen. Near the bottom of the shell the corrosion cells quickly polarize because of the excess water. Near the surface of the oil less polarization occurs because of the greater supply of oxygen. Needless to say, any additional agitation from mechanical mixers or from normal filling and emptying operations tends to promote corrosion by bringing fresh supplies of both water and oxygen.

The temperature of the shell while submerged has an important effect on the corrosion rate found in the lower courses. Tanks which are situated so that they are exposed to solar radiation all day will usually have the most severe corrosion on the south and east sides. Often the southeast corrosion rate on the bottom half of the tank will be twice that found on the north side of the tank in the lower courses.

Floating Roof Tanks

Completion of the corrosion reaction does occur on the upper courses of floating roof tanks. Beginning at the top of the shell it is found that the scale is thin and dry. The color is a dark red-brown (Fe_2O_3). Progressing downward, the color of the scale lightens to a reddish tan color and the quantity increases. At about mid-height the scale builds up to ⅛ inch to ¼ inch thick. But, due to the scraping action of the roof shoes, most of it sloughs off leaving only patches of scale. As soon as the shell is submerged for a little while the light colored scale begins to build up again.

In the bottom third of the tank the scale is continuous and has about the same texture and thickness as that found in the lower part of cone roof tanks in the same service and the same location. The corrosion rate in this area of gasoline tanks is about the same (4 to 7 mpy) as in cone roof tanks. In floating roof tanks the corrosion reaction takes place in the same sequence as cone roof tanks except that when the shell is exposed there is an unlimited supply of oxygen and more conversion of the scale to Fe_2O_3. While the shell is submerged much less air is supplied from the vapor space which is very small as compared to a cone roof tank. However, the vapor space, which is between the periphery of the roof and the shell, supplies air to the oil next to the shell which is diffused downward along the shell at night when the air is pulled into the vapor space. Thus the oxygen may be supplied into the scale at nearly the same rate as for cone roof tanks even though the oxygen concentration in the total volume of oil may be considerably smaller.

TABLE 3—Plate Thickness for 90 Foot x 48 Foot Tank*

	SHELL COURSE					
	1 (top)	2	3	4	5	6
API Design	0.250″	0.250″	0.313″	0.422″	0.531″	0.625″
Required for Water	0.092″	0.197″	0.302″	0.407″	0.511″	0.616″
Required for Gasoline	0.068″	0.146″	0.223″	0.300″	0.378″	0.455″
Corrosion Allowance Gasoline	0.182″	0.104″	0.090″	0.122″	0.153″	0.170″
Remaining Life—Years*	23	13	10	20	25	28
Required for 20 Year Life*	0.228″	0.306″	0.383″	0.420″	0.498″	0.575″
Final Design	0.250″	0.313″	0.391″	0.422″	0.531″	0.625″

* The plate thickness required for 20 year life and the remaining life for the original design are calculated for corrosion rates of 8 mpy for the top 3 courses and 6 mpy for the bottom 3.

Corrosion Allowance

The cost of corrosion in storage tanks can be reduced by using additional corrosion allowance in the design so that all courses in the tank will have about the same life. The first course of cone roof tanks to reach the minimum allowable plate thickness are of course, those in the area of highest corrosion. However, in checking the corrosion allowance provided by the recommended API design for all-welded tanks[8] it is found that the courses subject to the highest corrosion rate have the lowest corrosion allowance. The API recommended design thicknesses are calculated for the strength required when the tank is filled with water. If the minimum allowable plate thickness is calculated for the tank filled with gasoline (60 degrees API—Specific Gravity 0.7389) a corrosion allowance of about 26 percent of the original plate thickness is provided.

Table 3 illustrates the design and required thicknesses for a 90 foot x 48 foot gasoline tank. If the corrosion rate is assumed to be 8 mpy for the top three courses and 6 mpy for the lower three, the remaining life for the second and third courses in the recommended design is 13 and 10 years respectively while the other courses have remaining lives of 20 to 28 years. By making a slight increase in the design thickness for these courses, their anticipated life can be increased to 20 years thus giving a better balanced tank design without materially affecting the cost of the tank.

Corrosion Control

Since water and oxygen must both be present for the corrosion reaction to occur, any system which excludes or reduces one or the other should also reduce corrosion as well. Water would be very difficult to exclude from the tank because even though the bottom water layer was not used the absorbed water from processing would still be present. On the other

hand, the amount of oxygen can be materially reduced by pressure storage, gas blanketing, inhibition or by some types of breathing systems. In liquefied petroleum gas spheres and storage drums for instance, the corrosion rate is low (1 mpy to 3 mpy) because there is no breathing. Connate water is present in these drums from processing and must be removed periodically. However, since there is no breathing the only oxygen present is the very small amount which can carry through the processing equipment with the L P G.

Gas Blanketing

Cone roof gasoline tanks in natural gasoline service (15-18 psi Reid Vapor Pressure) are a good example of one type of gas blanketing. The corrosion rate is low (1 mpy to 3 mpy) because the amount of oxygen breathed in from the atmosphere is materially reduced due to the gas blanketing effect produced by the evaporation of the stock. During the night when the temperature drops low enough for inbreathing normally to occur, the negative pressure created by the breather valve undoubtedly promotes continued evaporation from the oil surface and materially reduces the amount of air drawn in.

Inhibitors

Inhibition of gasoline storage tanks with an oxygen absorbing inhibitor does not appear feasible. In pipe lines the only temperature change is a slow cooling of the oil and there is no addition of oxygen except possibly at intermediate pumping stations.[1] Tanks have daily temperature changes, at least on the shell surface and in the vapor space, and daily replenishment of oxygen from the air breathed in. Therefore, periodic inhibitor additions and mixing would be required for each batch of oil to maintain the concentration.

Breathing Systems

The use of closed breathing systems for cone roof tanks in gasoline service should reduce corrosion provided they are designed so as to minimize the amount of oxygen drawn into the tank. Normally, vapor recovery systems are designed to gather and recover gasoline vapors, which are usually vented to the atmosphere, to prevent stock losses and air pollution. Since these systems usually work at a slightly negative pressure they would tend to increase rather than decrease the amount of air drawn into the tank. One group of gasoline tanks which has been tied into a vapor recovery system for a number of years did not show any reduction in corrosion rate below that of other tanks in the same area and the same service.

Breathing systems which use a balloon or gas holder to store the excess vapor and then return it to the tanks as needed are designed to operate at a slight positive pressure most of the time. Therefore, they could be expected to reduce the amount of air drawn into the system and consequently the corrosion.

Protective Coatings

Protective coatings have been used in an attempt to reduce corrosion in existing gasoline storage tanks with results varying from good to bad. Gunnite has been applied to the internal surfaces of both cone and floating roof tanks for corrosion protection. A large number of gasoline tanks which had one or more courses approaching the minimum allowable thickness, have been gunnited with good results. The size of the tanks varied from 30 feet x 10 feet up to 150 feet x 48 feet both for the cone and floating roof type.

J. S. Cook[9] reports excellent protection of run down pans and tanks handling sour crudes with gunnite. With proper preparation of the shell the gunnite adheres tightly and even though it develops minute cracks there is no apparent corrosion. The cracks allow seepage of oil to the metal surface but apparently there is not enough circulation to cause any appreciable corrosion.

A number of paints have been tried on the internal surfaces of both cone and floating roof tanks with rather poor results. In cone roof tanks and on the lower 10 feet of floating roof tanks the paints have failed rapidly. Apparently during the periods of submersion in the oil, paints fail much more rapidly than they do in atmospheric exposures. Paints on the upper portion of floating roof tanks have the same life as for most atmospheric applications. However, the roof shoes scrape the paint off where they rub on the shell and these areas continue to corrode at the usual rate.

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Any discussions of this article not published above will appear in the December, 1957 issue.

Part 2 of 2 Parts

The Corrosion of Iron in High-Temperature Water*

Part 2—Kirkendall Experiments

By D. L. DOUGLAS* and F. C. ZYZES**

Abstract

Kirkendall-type experiments were carried out in order to identify the ion species diffusing through the magnetite film formed on corrosion of iron in high temperature water. Iron disks were painted with a slurry of $Ni^{63}O$, and the surface activity was measured. After corrosion the activity decreased. This decrease is shown to be most readily explained by a covering of $Ni^{63}O$ by magnetite formed as iron ions diffuse outward from the metal-oxide interface to the oxide-water interface. 4.6.5

Introduction

SINCE THE original work by Kirkendall[1] and later by Smigelskas and Kirkendall[2] the "Kirkendall" or "marker" technique has been used by many investigators to identify the species diffusing across a phase boundary. With more refined techniques the method may be used to measure diffusion rates. The work of Himmel, Mehl, and Birchenall[3] is an example of the latter.

Although it appeared that ionic diffusion was involved in the oxidation of iron by high-temperature water, the question as to whether the iron ion or the oxygen ion was diffusing at the greater rate was unanswered. Since most of the oxide must be magnetite, it was considered more likely that iron ions were the principal diffusing species. In an attempt to obtain an unequivocal answer to the question, two series of Kirkendall experiments were carried out.

In the first series, coupons of Armco iron were partially covered with an inert marker and then exposed to deoxgenated and deionized water at high temperature. Aluminum oxide was used as the inert marker, and its position in relation to the interfaces, iron-oxide and oxide-water, gave a qualitative indication as to which ionic species has greater mobility in the process by which iron is oxidized by water.

Since the crude type of marker experiment may be open to some objection and because inordinately long exposure times are required for a clear indication, a more refined experiment, which permitted a semi-quantitative measure of the corrosion rate, was undertaken using Ni-63 as a radioactive marker. The choice of Ni-63 was dictated by the fairly unique properties of the emitted radiation. Ni-63 emits a weak beta particle with a maximum energy of 62 kilovolts.[4] Using the formula of Katz and Penfold[5] the range in aluminum is 5.6 mg/cm². The half-thickness in aluminum should be approximately one-tenth the range,[6] or about 0.5 mg/cm². Since the average atomic number of magnetite is 16, its behavior in absorbing and scattering electrons is about the same as that of aluminum. The corrosion films produced after exposing iron for a few hundred hours in high-temperature water absorb a measurable

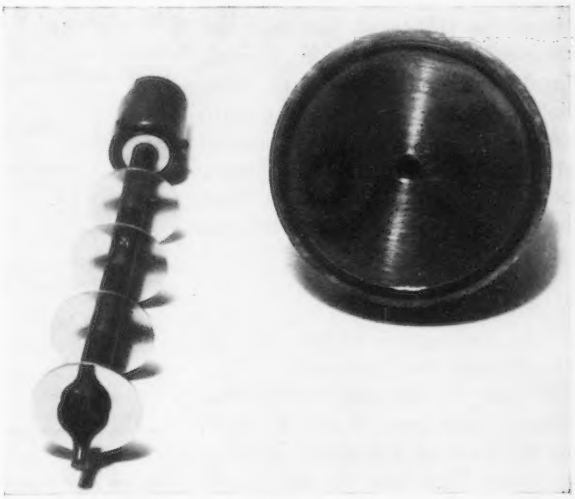

Figure 1—Kirkendall experiment assembly. Four iron disks are shown mounted on the iron bolt. The stainless steel holder contains an Al_2O_3 spacer to insulate the bolt and disks from the autoclave. The hole in the bottom of the autoclave lid is threaded to receive the holder assembly.

TABLE 1—Aluminum Oxide* Marker Experiments

Experiment	Temp. (Degrees C)	Time (Hours)	Observations
1	360	1054	Al_2O_3 completely covered by magnetite. Original pattern visible.
2	180	816	Al_2O_3 not covered. Tight black oxide film underneath.
3	180	1344	Al_2O_3 still visible and could be brushed off easily. Tight black oxide film underneath.

* Average diameter of Al_2O_3 particles determined by microscopic observation to be 0.8 to 1.6μ.

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fraction of the beta particles emitted from Ni-63 at the iron-oxide interface. Thus, it was possible to distinguish rather easily between the two possible cases, iron ion diffusion and oxygen ion diffusion, by following the change in surface activity or iron coated with a layer of Ni-63.

Experimental

Iron

Disks were punched from the Armco iron stock described in Part 1. Before punching, the material was polished by techniques also detailed in Part 1. The disks were ⅞ inch in diameter, a size which just fits the sample holder of a standard windowless flow-counter.[1] A 3/16-inch hole in the center of each disk permitted mounting several (separated by appropriate iron spacers) on a 10-32 bolt. The bolt was held rigidly along the axis of the autoclave, but insulated from the stainless steel autoclave (identical to those described in Part 1) with an alumina insulator. Figure 1 shows the disk-holding arrangement along with an autoclave cover. The threaded stem of the holder is drilled out to permit evacuation of the gas space in the autoclave through the gas port in the cover.

Nickel-63

The radioactive nickel was obtained from the Radioisotope Sales Division of the Oak Ridge National Laboratory. It was obtained in the form of a solution of $NiCl_2$ in dilute HCl, and the specific activity was 13.4 mc/gm. The specific activity was reduced to the desired level by adding reagent grade nickel chloride. Nickel oxide was prepared by precipitating the hydroxide and igniting. The oxide was ground in an agate mortar to reduce the particle size as much as possible. No measure of the particle size was made. That the radiochemical purity was

[1] An example of a windowless flow counter similar to the one used in these experiments is the "Preflush Flow Counter," sold by Radiation Counter Laboratories, Chicago, Illinois. Counting was done in the proportional region with methane as the counting gas. Power supply and scaler were supplied by RCL in the form of their "Nucleometer."

TABLE 2—Experiment No. 1

Hours (Accumulated)	Disk No. 1		Disk No. 2		Disk No. 3		Disk No. 4		Scale Thickness From Hydrogen Evolution (mg/dm²)
	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	
119	0.776	19	0.666	31	0.822	15	0.822	15	20—32*
232	0.758	21	0.610	36	0.752	22	0.762	21	24—41
471	0.700	27	0.536	48	0.680	30	0.686	29	29—50

* The quoted range of values covers the spread in the hydrogen evolution corrosion measurements.
Note: Disks were emery-polished in air through No. 240 paper; corrosion temperature was 360 C

TABLE 3—Experiment No. 2

Hours (Accumulated)	Disk No. 1		Disk No. 2		Disk No. 3		Disk No. 4		Scale Thickness From Hydrogen Evolution (mg/dm²)
	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	
4.5	0.964	2.8	0.764	21	0.970	2.3	0.900	8.1	5.8—8.7
26	1.110	..	0.729	24	0.931	5.4	0.867	11	12—19
92	1.053	..	0.681	29	0.853	12	0.796	17	17—29
180	0.869	11	0.657	32	0.816	16	0.770	20	23—37
339	0.777	19	0.638	34	0.769	20	0.754	22	27—45

Note: Disks emery-polished under kerosene through No. 400 paper and annealed in hydrogen at 820 ± 10 C for 1 hour; corrosion temperature was 360 C.

TABLE 4—Experiment No. 3

Hours (Accumulated)	Disk No. 5		Disk No. 6		Disk No. 7		Disk No. 8		Scale Thickness From Hydrogen Evolution (mg/dm²)
	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	
4.8	0.946	4.2	0.830	15	0.806	17	0.739	23	5.8—8.7
18	0.908	7.4	0.788	19	0.742	23	0.625	36	10—16
83	0.857	12	0.671	30	0.637	35	0.488	55	17—29
171	0.815	16	0.608	38	0.604	39	0.634	63	23—37

Note: Disks emery-polished under kerosene through No. 400 paper; corrosion temperature was 360 C.

TABLE 5—Experiment No. 4

Hours (Accumulated)	Disk No. 9		Disk No. 10		Disk No. 11		Disk No. 12		Scale Thickness From Hydrogen Evolution (mg/dm²)
	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	
22	1.51	..	1.34	..	0.986	1.1	0.982	1.4	5.8— 8.7
111	1.52	..	1.32	..	0.978	1.7	0.967	2.6	7.5—10.4
273	1.50	..	1.29	..	0.962	3.0	0.932	5.4	10.4—13.9

Note: Disks electrolytically polished; corrosion temperature was 240 C.

TABLE 6—Experiment No. 5

Hours (Accumulated)	Disk No. 17		Disk No. 18		Disk No. 19		Disk No. 20		Scale Thickness From Hydrogen Evolution (mg/dm²)
	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	A/A₀	d (mg/dm²)	
22	0.862	11	0.841	13	0.869	11	0.841	13	5.8— 8.7
111	0.799	17	0.777	19	0.799	17	0.783	19	7.5—10.4
273	0.790	18	0.755	22	0.786	18	0.761	21	10.4—13.9

Note: Disks emery-polished under kerosene through No. 400 paper; corrosion temperature was 240 C.

sufficient for these purposes was established by taking an aluminum absorption curve in the flow counter. This was done with a sample mounted on a glass disk and one mounted on an iron disk; both mounts gave a half-thickness of 0.53 mg Al/cm².

Method

A water slurry of the $Ni^{63}O$ was made up such that the iron disks, when about one-half covered with this mixture, would register over 10,000 counts per minute. The slurry was dried in an oven at 100 C, and any loose oxide was brushed off before counting. In each experiment four disks punched from the same polished strip, were mounted on the bolt. The autoclave was filled with a quantity of deionized and deoxygenated water which would cover at least three of the disks at the selected corrosion temperature. After the autoclave had been quickly sealed, the gas space was evacuated and then filled with helium through the gas port in the lid. The port was closed with a plug. The air in the gas space was replaced by helium in order to be sure that the oxidation of the iron was due to reaction with water and not with oxygen. As a further precaution the filling operations were carried out in a gloved box whose atmosphere contained not more than 1 percent oxygen.

Heating of the autoclaves was carried out in the same furnaces used in the corrosion experiments described in Part 1. After removal from the furnace and cooling, the autoclaves were opened; the disks were carefully rinsed with distilled water and then with reagent grade acetone and dried in air. The disks were stored in a desiccator until counted.

Since the possibility existed that some of the $Ni^{63}O$ was washed off into the water in the autoclave, the water, combined with that used to rinse the disks, was acidified with HCl and evaporated to a small volume. An aliquot of this volume was evaporated to dryness on a ⅞-inch circular glass microscope slide cover. This was counted on the flow counter. In no case was more than 5 percent of the total activity (total of all four disks) found in the autoclave filling water and washings. Further assurance that there was no leaching or washing away of the $Ni^{63}O$ was obtained by dissolving the iron disks and separating the nickel by extraction of the dimethylglyoxime and complex into chloroform (the assistance of E. Shirley of the Metallurgical and Chemical Analysis Activity is gratefully acknowledged here). Aliquots of a solution of $NiCl_2$, prepared by destroying the complex in HCl, were counted on the flow counter. Uncertainties in the counting technique—principally involved in the preparation of samples for counting in a reproducible way—made it impossible to calculate from this the precise amount of NiO which had been washed off into the water. However, the results established unequivocally that the observed decreases in activity of the disks could not be accounted for by this mechanism.

Results

The results of the aluminum oxide marker experiments are given in Table 1. Two coupons were used in each experiment; each was polished under kerosene through No. 400 emery paper.

Radioactive marker experiments were carried out at 240 C and at 360 C. Five runs of four disks each were made. With three exceptions the observed beta particle emission from the disks decreased with increasing length of exposure to the high-temperature water. In the three anamalous cases the activity first increased and later decreased with corrosion time. Since all of these exceptions involved the uppermost two disks, which were subject to some washing action as the water expanded on heating the autoclave, it is believed that the initial increase in observed activity was due to a more uniform spreading of the slurry over the disks before any significant amount was covered by iron oxide. In general, the agreement among disks was good, and that between the lower two disks was excellent.

From the observed decreases in activity it was possible to calculate a scale thickness by using the expression:

$$A/A_o = e^{-\frac{\mu}{p}d} \quad (1)$$

where:

A_o = beta particle activity with no absorber or original activity.
A = activity measured after corrosion, hence under a certain thickness of cale.
μ/p = the mass absorption coefficient in dm²/mg
d = thickness of the absorbing layer in mg/dm²

The mass absorption coefficient is related directly to the half-thickness:

$$d_{1/2} = \frac{0.693}{\mu/p} = 0.53 \text{ mg/cm}^2 = 53 \text{ mg/dm}^2, \quad (2)$$

or:

$$\mu/p = 1.31 \times 10^{-2} \text{ dm}^2/\text{mg} \quad (3)$$

By a rearrangement of Equation (1) and substituting in the value of μ/p from Equation (3) the following equation is obtained:

$$d(\text{in mg/dm}^2) = \frac{-2.303 \log (A/A_o)}{1.31 \times 10^{-2}} \quad (4)$$

The results of the five experiments are given in Tables 2-6. Included in these tables are scale thick-

TABLE 7—Nickel Diffusion Test

Experiment*	Disk No.	Corrosion Temp. (Degrees C)	Corrosion Hours	A/A₀	Final Activity (Counts/Min.)	Activity After 90 Hours in Vacuum at 360 C
2	3	360	339	0.769	4843	4837
3	8	360	171	0.436	8960	8892

* Refers to original experiment number.

TABLE 8—Nickel-63 Recovery Experiments

Experiment	Disk No.	Corrosion Temp. (Degrees C)	Corrosion Hours	A/A₀	A (Recovered)/A₀
2	1	360	339	0.777	0.737
2	3	360	339	0.769	1.09
3	5	360	171	0.815	0.867
3	7	360	171	0.604	0.77
4	9	240	273	1.49	0.98
4	12	240	273	0.932	1.045
5	20	240	273	0.761	1.22

nesses calculated from the corrosion data of Part 1 assuming the corrosion scale to be magnetite with its bulk density. In line with the previous comments on the agreement among the results for any one experiment, the order of disks along the holder was always the lowest-number disk closest to the top of the autoclave; the remainder were placed in sequence toward the bottom. Corrections have been applied for the Ni-63 found in the corrosion water and washings.

In order to prove that the decrease in activity was not caused by a diffusion of nickel into the iron substrate, two of the corroded disks were heated to 360 C in a vacuum for several days. No change in activity resulted, as shown in Table 7. This result is what might be expected from calculations based on diffusion data in the literature.[7]

Nickel separations were carried out with several of the disks to establish that the Ni-63 had not been lost to the water and escaped detection. Preliminary experiments showed that the extraction efficiency varied between 80 and 100 percent. This fact, coupled with the difficulty encountered in making reproducible mounts, made it impossible to calculate the actual amount of activity lost. The results in Table 8 make it clear, however, that most of the observed decrease in activity with corrosion must be due to a burying of the $Ni^{63}O$ by a corrosion scale.

Discussion

The conclusion that is drawn from the above Kirkendall experiment data is that the oxidation of iron by water in the temperature range 240 to 360 C proceeds by diffusion of the iron ions outward from the metal to the water interface. In view of the fair agreement between the scale thicknesses calculated from the beta particle absorption equation and those calculated from the hydrogen evolution data, it seems likely that oxide ion diffusion does not take place to an appreciable extent. This is in accord with the observations of Davies, Simnad, and Birchenall[8] that diffusion in magnetite at higher temperatures is predominantly cationic. It is interesting that at the lower temperature of 240 C, where the hydrogen evolution data showed that the rate-determining step is most likely to be an interface reaction, diffusion of iron ions remains an important step in the over-all reaction. The rate-controlling reaction must then take place at the oxide-water interface.

At this time there is very little information available about the reaction mechanism below 240 C. The inert marker results indicate that iron ion diffusion may not play a part. However, the evidence is very meager. The fact that the Al_2O_3 was not covered may simply reflect a very low rate of reaction and hence a film too thin to cover the particles.

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End of Part 2

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NACE TECHNICAL COMMITTEE REPORT

Hydrogen Absorption, Embrittlement And Fracture of Steel

A Report on Sponsored Research on Hydrogen Sulfide Stress Corrosion Cracking Carried on at Yale University, Supervised by NACE Technical Unit Committee T-1G On Sulfide Stress Corrosion Cracking[1]

By ARNOLD E. SCHUETZ[2] and W. D. ROBERTSON[3]

Foreword

THE FOLLOWING report, entitled "Hydrogen Absorption, Embrittlement and Fracture of Steel" by Arnold E. Schuetz and W. D. Robertson, presents a summary of the experimental data developed by and conclusions drawn from a research project on sulfide stress corrosion cracking sponsored by the National Association of Corrosion Engineers at Yale University. This project, which was administered by Unit Committee T-1G, was made possible initially by a grant from The International Nickel Company, Inc., and its continuation was assured by contributions from 20 additional companies.

The primary objective of the work was to develop basic information on the metallurgical factors involved in sulfide corrosion cracking of steels with the expectation that such fundamental knowledge would cast some light on the mechanism of the phenomenon and would serve as a basis for continuing work by others directed toward practical solutions of specific service problems.

The statements, opinions, and conclusions expressed in this report are those of the authors and are not necessarily those of the National Association of Corrosion Engineers or of its Unit Committee T-1G. Application of the information contained in the report to the many practical problems involved in handling fluids containing hydrogen sulfide should be made with caution. Written discussions of this report, particularly those presenting additional data on sulfide corrosion cracking, are solicited and will be published from time to time in CORROSION.

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The Atlantic Refining Company
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Introduction

Spontaneous failure of steel in corrosive media containing hydrogen sulfide has been observed in a number of instances.[1-4] About 1950 the problem assumed serious proportions in the petroleum industry, as a result of failure of alloy tubing in a few sour gas-condensate wells. The serious character of the problem was not defined by the number of failures but by the magnitude of the economic and technical hazards implied by the failures. A single fracture

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Abstract

Results of a four-year research program at the Yale University Metallurgy Department are presented. After a review of the problems associated with spontaneous fracture of steels in sour gas condensate wells an account of the investigation is given as follows:

Part 1—Hydrogen sulfide embrittlement was compared with that caused by cathodic charging. An investigation was made of spontaneous fracture under conditions of constant deformation, as dependent on alloy composition, heat treatment, plastic deformation and chemical environment.

Part 2—Hydrogen absorption by iron-nickel alloys from hydrogen sulfide was compared to that from sulfuric acid.

Part 3—Permeation rate of hydrogen through austenitic, martensitic and ferritic states of iron-nickel alloys was measured.

Part 4—Time dependence of static fracture of iron-nickel alloys as a function of applied stress, structure and hydrogen concentration was evaluated.

The following conclusions are given: Basic cause of failure is absorption of hydrogen. Principal factors in service failure are internal stress, magnitude of applied stress and hydrogen content. Both martensitic and ferritic structures fail when the stress level and hydrogen content are sufficiently high. Trace amounts of carbon, oxygen and nitrogen in 10 percent nickel steel have no detectable effect on failure. Adoption of limiting strength or hardness below which fracture does not occur in constant deformation tests is unreliable in predicting service life.

Because it is impractical to lower operating stress significantly below apparent "endurance limit" the remaining alternative is reduction of hydrogen concentration in the steel. Deadload tests in the environment must be made before safe operating procedures can be defined. 3.2.2

of casing or tubing in high pressure wells could lead to loss of a well and, possibly, extensive damage to the formation; furthermore, unpredictable failure of materials could seriously impede the development of sour gas-condensate fields in Canada and the United States.

Early recognition of the problem led to extensive laboratory and field investigations by the operating companies and to the organization, within the National Association of Corrosion Engineers, of Technical Practices Committee TP-1G to coordinate field tests and operating experience. A summary of laboratory and field investigations was published in 1952.[5] The complexity of the problem and numerous mechanisms of failure proposed in this symposium indicated that more extensive investigation was required.

It was anticipated that effective remedial measures could be taken only when and if a reasonably complete understanding of the mechanism was available; further, specification and interpretation of laboratory testing procedures also depended on a detailed knowledge of mechanism. Accordingly, in 1951, Committee TP-1G initiated research in the Metallurgy Department at Yale University for the purpose of investigating the mechanism of failure of steel in hydrogen sulfide.

This report contains a summary of the experimental data and conclusions drawn from the work conducted at Yale University from 1951 to September 1955.

A number of documented hypotheses regarding the probable cause of failure had been advanced before the present investigation.[5] In order to place subsequent work in proper perspective, it is necessary to review, briefly, the available information and the proposed mechanisms.

Hypotheses Reviewed

The term "sulfide corrosion cracking" reflects some uncertainty with regard to the basic cause of failure. This term was chosen because there was lack of agreement on whether failures were primarily caused by the effects of hydrogen absorbed from the corrosive media or were the result of structural corrosion and associated stress corrosion cracking. The difficulty of deciding between these two basic causes is inherent in the problem; failure is the ultimate criterion and, because either phenomena may produce failures in similar circumstances, a clear separation is difficult to obtain. However, a definite conclusion is absolutely necessary with respect to the two proposed causes of failure because the remedial measures may be quite different in the two cases. Accordingly, one of the objectives of the present work was the identification of the principal cause of failure in terms of the effects of hydrogen or stress corrosion cracking.

Because failures generally occurred in alloy steels, various hypotheses have been advanced regarding metallurgical structures possibly responsible for failure: 1. That a martensitic network[5] was the path of failure and, 2. That transformation of retained austenite resulting from applied stress was a cause of failure. The first proposal was based on detailed metallographic observations and the apparent beneficial effects of normalizing heat treatments designed to eliminate untempered martensite. The mechanism based on the transformation of retained austenite[6] considered the fact that the solubility of hydrogen in austenite at one atmosphere pressure is greater than in ferrite, extrapolated to the same temperature, and when the austenite transformed under stress the hydrogen could be precipitated in the resulting ferrite (martensite) at a very high pressure and thus could cause failure. Although both proposals had some foundation in theory and experimental data, whether or not either was a necessary and sufficient condition for failure remained to be proved.

Mechanical Properties Critical

Associated with the structural and composition dependence of failure was frequent observation that failures were dependent on mechanical properties. Specifically, failures seldom occurred below a Rockwell hardness of C-20; more recently, this lower limit has been raised to C-25.

This conclusion, however, regarding dependence of failure on mechanical properties was drawn from laboratory investigations using constant deflection testing procedures in which the magnitude of the applied stress (and elastic energy) is limited by mechanical properties and specimen geometry. For thin specimens generally used, the tensile stress falls rapidly to zero at the neutral axis of a bent beam and relaxation phenomena operate to reduce the internal stress. Thus, absence of failure of lower strength materials in a constant deflection test is not necessarily conclusive evidence that failure cannot occur under loading conditions which maintain a constant stress over the entire section. Accordingly, predictions regarding service life, based on constant deflection tests, may be limited to the corresponding state of stress in a structure and the data are inadequate for prediction under the usual conditions approximating constant load and stress.

Available data regarding dependence of susceptibility on alloy composition do not provide definite information from which to derive conclusions, either with respect to mechanism or relative susceptibility. To some extent the inconclusiveness of the evidence is a consequence of constant deflection testing conditions which do not permit a separation of properties and applied stress; but, in any case, a systematic study of alloy composition has not been made. Extensive testing of commercially available material used in the petroleum industry has demonstrated that austenitic steels and nickle-base alloys are essentially immune; that high nickel steels are extremely susceptible and that the widely used low alloy tubing materials are more or less susceptible, depending on stress and chemical environment. Effects of small, specific additions, either deliberately added or when present as impurities, are unknown, though there is limited evidence that some additions may be beneficial.

As a result of work reported in the previous symposium,[5] there is little doubt that hydrogen sulfide is the principal agent promoting fracture. Further, Bastien and Amiot[4] have shown that embrittlement does not occur in the absence of water or in a non-ionizing medium such as benzene. Fraser and Treseder[5] have shown that the addition of acetic acid to an aqueous solution promotes fracture and lowering the test temperature from 25C to 0C also appears to increase fracture susceptibility.

Basic Decision Not Made

The basic decision between stress corrosion cracking or failure associated with hydrogen absorption was not settled when the present project was initiated. Experiments showed that most, if not all, alloys susceptible to failure in a sulfide environment spontaneously failed when cathodically charged with hydrogen, provided sufficient static stress and/or cold work was applied. Indeed, under these conditions, failure incidence assumed alarming proportions. Against a mechanism involving hydrogen, there was evidence that reproducibility of failure was poor in specimens cathodically charged under constant deformation conditions. Furthermore, tests with beam specimens[5] covered with a vinyl coating opposite the area of maximum stress, failed outside the coated area at a position of lower stress. This experiment was interpreted to mean that a corrosion process was involved because it was assumed hydrogen concentration was uniform throughout the specimen. Likewise, in some tests cathodic reduction of hydrogen from sodium hydroxide did not cause failure and this was interpreted as evidence that hydrogen alone was insufficient to cause spontaneous failure in a sulfide environment.

Corrosion Cracks Found

Finally, structural evidence of corrosion cracks has been found. In particular, it was frequently observed that cracks originated at the base of pits produced by localized corrosion. However, it was observed also, by Fraser and Treseder,[5] that failure in a sulfide environment was characterized by a single macroscopic crack while in a cyanide solution multiple cracking of

the same alloy was observed. Thus, it appeared that cracking in a sulfide environment could, to some extent, be distinguished from typical corrosion cracking.

Plan of Investigation

In order to separate the numerous variables and evaluate their relative contribution to fracture of alloys in hydrogen sulfide, it was concluded that the present study should be confined to the simplest alloy system which exhibits the phenomena. The system chosen was a set of relatively pure iron-nickel alloys, varying from zero to 30 percent nickel; as indicated later, the carbon content also was considered in iron-carbon alloys.

The iron-nickel system is unique in that all three structures characteristic of ferrous alloys may be obtained with a minimum of two different compositions. Figure 1 is a combination of the most recently determined iron-nickel equilibrium diagram[7] with the martensite transformation temperature, Ms, and the temperature at which the martensite transformation ceases on cooling, Mf; data for the two transformation temperatures were obtained from the experimental study of Kaufman and Cohen.[8]

Slow cooling of a 10 percent nickel alloy from above the γ—α/γ transformation temperature produces body centered cubic ferrite; quenching from the same temperature produces a nickel martensite of the same composition. The 30 percent nickel-iron alloy is metastable face-centered cubic austenite at room temperature; cooling to —190C in liquid air transforms about 95 percent of the austenite to martensite of the same composition. Thus, all three structures can be obtained at room temperature in two alloys, with martensitic structure common to both alloys. It was anticipated that this series of alloys would provide data indicative of the significance of the various structures and, of the relation of composition to failure.

Evaluation of hydrogen sulfide as an embrittling medium was necessary in order to obtain a measure of the magnitude of the effect and the range of chemical conditions in which it occurs. To establish a basis for comparison and determine the range of operation for subsequent work, embrittlement in hydrogen sulfide was compared with corresponding phenomena resulting from cathodic charging of hydrogen from dilute sulfuric acid. Evaluation of embrittlement was made in terms of three observable properties:

1. The change in ductility measured by a bend test,
2. The effect of hydrogen on the yield point of iron and
3. Recovery of ductility in time.

The phenomena of recovery was considered specially significant in providing some measure of differentiation between a phenomenon involving hydrogen and one primarily associated with corrosion cracking, because, in corrosion cracking, structural damage would not be recoverable. In fact, this is one of the few phenomena distinguishing the two processes.

Extensive Fracture Tests Necessary

Extensive fracture tests were necessary to determine the significance of structural and compositional variables. To cover the wide range of conditions involved and to correlate the work with preceding investigations, a constant deformation test was used to disclose phenomena of major significance and to assist in evaluating the relative importance of different variables. Subsequent work at constant applied stress gave the necessary differentiation between mechanical properties and stress.

Because interpretation of the above fracture tests largely depended on knowing the hydrogen concentration under different structural and chemical conditions, it was necessary to measure hydrogen concentration in the alloys in a parallel set of experiments. Analytical work was essential because a literature review clearly indicated that complexities associated with hydrogen absorption are such that it is difficult to make predictions about hydrogen concentration and, further, the effect of structure in conditions under investigation was almost unknown.

Finally, a quantitative study of the fracture process under well defined conditions of stress and hydrogen concentration was required to give data helpful in interpreting phenomena observed under more complex conditions and which would provide material for a more general conclusion regarding the failure process.

Experimental work may be subdivided as follows:

Part 1—**Embrittlement and Cracking Phenomena**

1) Comparison of hydrogen sulfide embrittlement with that caused by cathodic charging of hydrogen.
2) Investigation of spontaneous fracture under conditions of constant deformation, as dependent on alloy composition, heat treatment, plastic deformation and chemical environment.

Part 2—**Hydrogen Absorption in Iron-Nickel Alloys**

1) Quantitative measurement of hydrogen absorbed by iron-nickel alloys from aqueous hydrogen sulfide as dependent on time, alloy composition and structure.
2) Absorption of hydrogen, cathodically discharged from sulfuric acid, as dependent on alloy composition, structure and chemical variables associated with the discharge process.

Part 3—**Hydrogen Permeability of Austenite, Martensite and Ferrite in Iron-Nickel Alloys**

1) Measurement of permeation rate of hydrogen through iron-nickel diaphragms in different structural states.

Part 4—**Delayed Fracture of Iron-Nickel Alloys**

1) Measurement of the time dependence of static fracture of iron-nickel alloys as a function of applied stress, structure and hydrogen concentration.

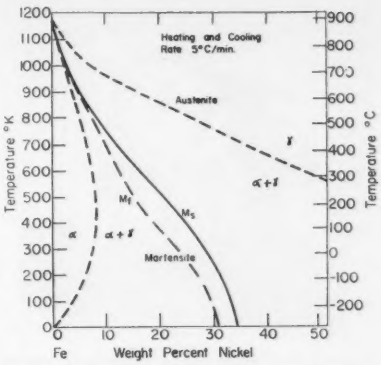


Figure 1—The iron-nickel system after Owen and Liu[7] with low temperature boundaries by Kaufman and Cohen.[8]

2) General conclusions regarding the fracture process.

For clarity and convenience each of the four principal subdivisions will be treated separately.

Experimental Procedure

Experimental procedure applicable to all work will be considered here while specific techniques employed for the separate phases will be treated under the appropriate subdivisions.

Materials

Two principal classes of materials were used. The composition of these alloys is given in Table 1.

The four alloys (A, AA, K and L) provided a means of studying the effect of carbon content and the accompanying structures produced by different heat treatments. Nickel alloys (B, E and G) were used to investigate the effect of nickel content and the different crystallographic structures obtainable in them.

Alloy N was used to determine the effect of titanium as an alloying element and as a means of removing traces of carbon, oxygen and nitrogen.

All materials, except those used for constant stress fracture experiments, were obtained in the form of cold rolled sheet, 0.0625-inch thick. The low carbon steel A was commercially available "magnetic" iron, equivalent to Armco iron and it is so designated in this report. All other materials were prepared by The International Nickel Company from Armco iron and electrolytic nickel. They were melted in an induction furnace and deoxidized with silicomanganese and 0.05 percent aluminum. Ingots were hot forged, rolled and finished by cold rolling 15 to 50 percent reduction in thickness.

Heat Treatment

Unless specifically excepted, all alloys were heat treated in a vertical, quartz-

TABLE 1—Composition of Alloys[1]

Mark	Ni	C	Mn	Si	S	P	Ti
A	—	0.023	0.09	0.014	0.033	0.013	....
AA	—	0.04	0.09	0.10	0.029	0.015	....
K	—	0.25	0.22	0.17	—	—	....
L	—	0.37	0.20	0.17	—	—	....
B	4.89	0.04	0.15	0.03	—	0.007	....
E	10.30	0.08	0.07	0.11	0.01	0.004	....
G	30.70	0.06	0.13	0.05	0.005	0.002	....
N	10.16	0.05	0.24	0.14	—	—	0.44

[1] Composition is given in weight percent.

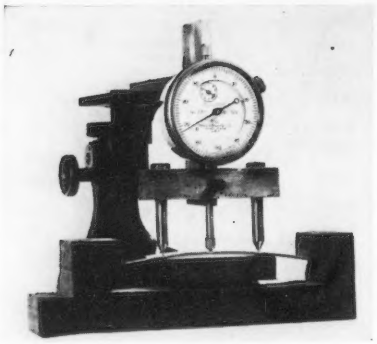


Figure 2—Dial gauge fixture arranged to measure curvature of strip specimens stressed by four-point loading.

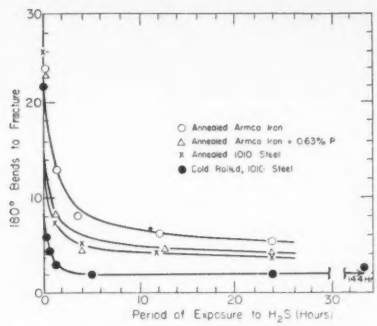

180° Bends to Fracture
Period of Exposure to H_2S (Hours)
Annealed Armco Iron
Annealed Armco Iron + 0.63% P
Annealed 1010 Steel
Cold Rolled, 1010 Steel

Figure 3—Change in ductility of Armco iron and mild steel resulting from exposure to hydrogen sulfide.

tube furnace in an atmosphere of purified argon. After placing specimens in the furnace at room temperature, the air was removed by a vacuum pump and the furnace refilled and evacuated several times, followed by filling with argon purified by passing it over titanium at 700C. Depending on the structure required, specimens were cooled either in the furnace to room temperature or were dropped from the furnace by a trigger mechanism into water or brine at room temperature.

As previously indicated, the iron-nickel alloys may be obtained in all three of the principal crystallographic structures associated with steel. Through the courtesy of Professor B. L. Averbach, of the Massachusetts Institute of Technology, a quantitative X-ray analysis was made of the 10 and 30 percent nickel steels after heat treatment. The 10 percent alloy was found to be completely martensitic ± 0.1 percent, when quenched in water from 1000C. The 30 percent nickel alloy was completely austenitic ± 0.3 percent, after cooling slowly to room temperature from 1000C. The indicated limits of uncertainty correspond to the sensitivity of the technique for a determination of ferrite and martensite in the two structures. Ferrite was obtained in the 10 percent nickel alloy by cooling slowly in the furnace; martensite was produced in the 30 percent nickel alloy (Ms temperature about —50C) by transformation in liquid air at —190C which produces a structure with about 5-10 percent retained austenite.[8]

Furnace cooled specimens had a clean surface that required no further treatment before testing. Quenched specimens had a blue temper-color film of oxide produced by brief passage through air and into the quenching water. Tests were conducted to determine whether mechanical or chemical removal of the film affected the results obtained in experiments on hydrogen absorption or embrittlement; no significant differences were found between specimens with or without films and all subsequent work was done with specimens taken directly from the heat treatment, without further surface preparation.

Experimental Procedure

Investigations involving exposure to saturated aqueous hydrogen sulfide were conducted in a 4-liter container at room temperature. Before beginning an experiment the system was purged of oxygen by tank nitrogen. Hydrogen sulfide from a cylinder was passed continuously through fritted glass dispersion discs into four liters of distilled water and the excess was absorbed in sodium hydroxide.

Specimens were supported in the container by fixtures cut from cloth-base Phenolite which, after an initial small expansion, proved extremely stable and convenient.

Constant deformation fracture experiments were conducted in the same apparatus, except that Phenolite fixtures were designed for the application of a bending stress by adjustable four-point loading. Apparent stress was calculated from the uniform curvature of the middle third of a 5-inch long specimen measured by a three point gauge of which one point was the active element of a one-thousandth inch dial gauge. The fixture and curvature measuring apparatus are shown in Figure 2.

Cathodic charging with hydrogen was performed in one liter beakers. The source of current was one or more six volt storage batteries, together with the necessary controlling and measuring apparatus. Platinum anodes were used invariably. For most experiments involving cathodically charged hydrogen, a 4 percent solution of sulfuric acid was used as electrolyte; but in special experiments a 10 percent solution of sodium hydroxide was used. In all cases, reagents were dissolved in distilled water. Unless otherwise indicated, cathodic current density was 2 ma/in.^{2} a value chosen as a result of a comparison of effects of hydrogen sulfide and cathodic charging. However, it developed that hydrogen concentration at saturation obtained with 2 ma/in.^{2} in a "poisoned" electrolyte is much larger than that absorbed by the alloys from aqueous hydrogen sulfide.

The cathodic poison employed was several drops of phosphorus dissolved in carbon disulfide.[9] As a result of previous work on embrittlement[10] it was anticipated that the use of this poison would improve reproducibility; however, later experimental work showed that phosphorus has no effect on hydrogen absorption and any improvement in reproducibility that can be attributed to the poison is questionable.

Part 1—Embrittlement and Fracture Phenomena

Bend Test Ductility

In order to evaluate the effect of hydrogen sulfide on ductility and, especially, on the extent of permanent corrosion damage, annealed Armco iron A and cold rolled mild steel were exposed to a saturated aqueous solution of hydrogen sulfide for periods extending to 140 hours and immediately tested. It was found that the most sensitive test was reverse bending of 1/16 inch specimens around a 1/2 inch mandrel. The number of 180 degree reverse bends required to produce failure was used as a measure of ductility and the testing procedure was surprisingly reproducible, usually better than 5 percent.

Results obtained are shown in Figure 3 as a function of the time of exposure to aqueous hydrogen sulfide. It is apparent that ductility falls rapidly to a low and **constant** value. At least for low carbon steel, damage is not progressive after the initial loss in ductility.

The shape of the curves is significant in that it appears to have the characteristics expected of a process dependent on diffusion—i.e., as a diffusion process the rate of penetration of hydrogen is proportional to the square root of time and, if ductility is inversely proportional to hydrogen concentration, the ductility should be inversely proportional to the square root of exposure time up to some limiting time at which the material is saturated. The data are generally consistent with this relationship and, because of subsequent hydrogen analysis, it is possible to explain the curves in some detail. Thus, ductility of annealed Armco iron diminishes continuously up to 24 hours and it has been found that hydrogen concentration, in this material only, does not reach a definite saturation limit in this time. On the other hand, the lower limit of ductility in cold rolled steel undoubtedly is associated with the subsequently observed saturation limit of hydrogen. Quantitative analysis is precluded by the complexity of the fracture process in bending and, in particular, by propagation of fracture inwards from the outer surface where hydrogen content reaches a maximum at an exposure time less than that required for saturation. Effect of surface hydrogen is apparent in the very rapid loss of ductility to a limiting value in the cold worked steel in about three hours, whereas complete saturation, as shown later for a 10 percent nickel steel, does not occur until about nine hours. In any case, for the materials and conditions, there is no evidence of intergranular corrosion or other time-dependent corrosion damage to the structure.

Further evidence that ductility changes are associated with hydrogen rather than structural damage is provided by an investigation of the recovery of ductility in time after exposure to hydrogen sulfide. Experiments were conducted with Armco iron and specimens were exposed to hydrogen sulfide for 1, 4 and 24 hours; following exposure specimens were allowed to age at room temprature, up to 70 days, or they were held in boiling distilled water for 1, 3 or 24 hours immediately following exposure to hydrogen sulfide. The results of aging at room temperature and at 100C are shown in Figures 4 and 5.

Evidently the recovery of ductility at 25C depends on the time of exposure to hydrogen sulfide and on aging time. Recovery is complete in 20 days for an exposure time of one hour and in 50-60 days for an exposure of four hours. After a 24-hour exposure, recovery is still incomplete in 70 days at 25C.

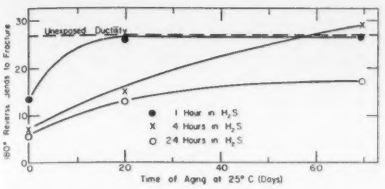


Figure 4—Recovery of ductility of annealed Armco iron at room temperature following exposure for 1, 4 and 24 hours to saturated aqueous hydrogen sulfide.

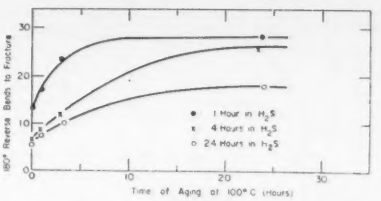


Figure 5—Recovery of ductility of annealed Armco iron as a result of aging at 100 C, following exposure of 1, 4, and 24 hours to saturated hydrogen sulfide.

Recovery at 100C follows a similar pattern, except at a higher rate. However, data obtained following treatment at 100C are complicated by the fact that the ductility of the steel itself changes as a result of treatment at 100C. Thus, aging at 100C for 24 hours increases the ductility of the unexposed material from 26 to 30 units. On the new basis, recovery after four hours' exposure to hydrogen sulfide is not quite complete in 24 hours at 100C and is certainly not complete in this time for material which was exposed for 24 hours.

To further evaluate the recovery process, specimens of Armco iron were exposed to hydrogen sulfide for 1, 4 and 24 hours and immediately immersed in boiling water for 1, 3 and 24 hours. Following this treatment at 100C, the specimens were aged at 25C and tested after 20 and 70 days. The results are shown in Figure 6.

First, it appears that recovery continues in time at 25C, after previous treatment at 100C. Secondly, the extent of recovery at 25C in a given time, say 70 days, depends on the extent of exposure to hydrogen sulfide and decreases with increasing exposure time. Third, as a function of time at 100C, the recovery passes through a minimum at one hour. This is shown in Figure 7 which is a rearrangement of the data for 70 days aging at 25C. Interpretation of these data is difficult owing to aging of the Armco iron independently of hydrogen. However, it is apparent that the recovery is directly affected by the hydrogen content after one hour at 100C because the magnitude of the minimum is different for different times of exposure to hydrogen sulfide for specimens having the same thermal treatment.

Yield Point Phenomena

The preceding results are indicative of the effects of hydrogen absorbed from hydrogen sulfide. To investigate these phenomena further, stress strain curves were obtained for Armco iron A exposed to hydrogen sulfide and comparative data were also obtained for the same material after cathodic charging at 2 ma/in^2 in sulfuric acid containing carbon disulfide. Two types of tensile specimen were used;

1. A rectangular strip, 1/2 x 1/16 x 5 inches, cut transverse to the rolling direction, and
2. Standard ASTM sheet tensile specimens cut parallel with the rolling direction.

The tensile test was conducted at 0.05 in/min., strain rate in a screw driven, recording machine. Tests were made at room temperature under two general conditions:

1. In air, 15 to 60 minutes after exposure and
2. In saturated hydrogen sulfide.

Results of exposure in hydrogen sulfide for 1, 4 and 24 hours and for cathodic charging in sulfuric acid are shown in Figure 8.

With respect to the carbon yield point, it is apparent that both the yield point elongation and the yield point stress are progressively reduced by exposure to either hydrogen sulfide or cathodic charging. Ultimate tensile strength and elongation are similarly reduced. Progressive lowering of yield point stress and elongation are very similar in appearance to the effect produced on the carbon yield point by increasing plastic deformation, such as employed in "temper-rolling" of steel sheet for deep drawing purposes.

To evaluate the role of hydrogen in suppressing the yield phenomena, similar tests were made after a recovery period of 70 days at room temperature, following exposure to hydrogen sulfide and to cathodic charging. Results are shown in Figure 9. Recovery of yield point stress and elongation is almost complete for a 1-hour exposure but it is not complete for the more extensive exposures, which is consistent with the observations on recovery evaluated by bend ductility.

Because the effect of elapsed time between exposure and testing in air was unknown, it was necessary to evaluate the phenomena under conditions of continuous exposure, so, standard tensile specimens were prepared with a metal container surrounding the gage section with the container closed at the top by a rubber membrane. The specimens and attached container were supported, without loading, in the tensile machine and the container was filled with saturated hydrogen sulfide. After 1 or 24 hours' exposure, the specimens were tested in the solution. Typical stress-strain curves are shown in Figure 10, together with corresponding data for specimens exposed for the same time and tested in air, a few minutes after removal from the solution.

Specimens with a reduced gauge section and cut parallel to the rolling direction had a much more pronounced yield point. When tested out of the solution, the yield point is not significantly affected, however, the yield point strain is completely eliminated when tested in the solution after the same exposure. Similarly after 24 hours a small residual yield strain appears when tested out of the environment. When tested in the solution after 24 hours, the yield point strain has disappeared, but a new phenomenon has appeared in the form of a discontinuous plastic strain distributed more or less over the entire range of elongation. Also, in the present case, the change in ultimate strength is small when compared with the transverse specimens.

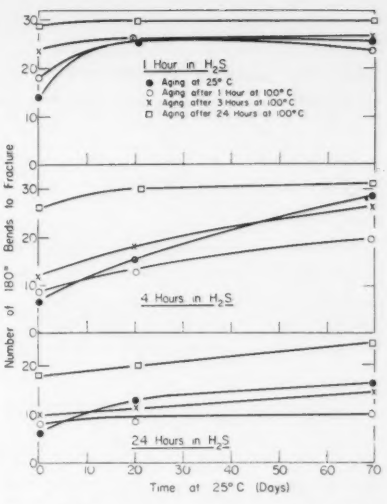

1 Hour in H_2S
Aging at 25° C
Aging after 1 Hour at 100° C
Aging after 3 Hours at 100° C
Aging after 24 Hours at 100° C
4 Hours in H_2S
24 Hours in H_2S
Number of 180° Bends to Fracture
Time at 25° C (Days)

Figure 6—Recovery of ductility of annealed Armco iron at room temperature following exposure of 1, 4, and 24 hours to saturated hydrogen sulfide and heating to 100 C.

With respect to the original purpose of these experiments, it appears that the effects of exposure to hydrogen sulfide and cathodic charging are qualitatively similar, both in the primary suppression of the yield point stress and elongation and in subsequent recovery.

The phenomenon itself is of considerable interest with regard to the mechanism by which hydrogen changes the mechanical properties of iron. After this work was completed and reported to Committee TP-1G in 1952, Cracknell and Petch[11] reported identical observations for a variety of steels when cathodically charged in sulfuric acid. H. C. Rogers[16] also has made similar observations and has extended the range of experimental data to the effect of temperature on the yield point suppression.

The basic cause of the yield point is now known and it lies in the pinning of dislocations.[12] At room temperature, the binding of carbon and nitrogen to dislocations is sufficiently strong to prevent the movement of dislocations until the upper yield stress is attained, after which dislocations are pulled away from their atmosphere of solute atoms and plastic deformation then continues at the lower yield stress characteristic of the unpinned dislocations. Rogers[13] has shown that hydrogen is similarly responsible for a yield point, but it does not appear above —120C. It seems, therefore, that the forces binding hydrogen to dislocations are much smaller than for carbon, approximately in the ratio of the absolute temperatures at which the yield points associated with hydrogen and carbon are observed to disappear, namely 150:700.[14] Furthermore, the rate of recovery of the yield point indicated in Figure 9, is more than an order of magnitude slower than that associated with the diffusion of carbon back to dislocations after plastic deformation,[15] where the rate of diffusion of hydrogen is several orders of magnitude greater than that of carbon. Accordingly, it appears that the effect of hydrogen on the yield point cannot be attributed simply to the displacement of carbon atoms from dislocations by hydrogen. A possible explanation may be found in the inhomogeneous stresses

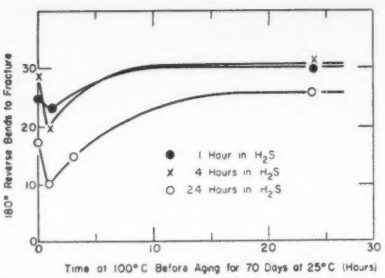
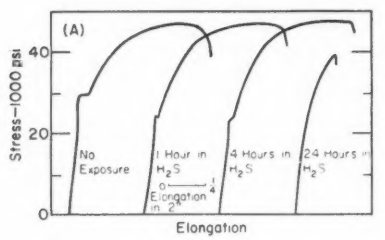


Figure 7—Recovery of ductility of annealed Armco iron after aging for 70 days at room temperature following exposure of 1, 4, and 24 hours to saturated hydrogen sulfide, and holding at 100 C for the indicated time.



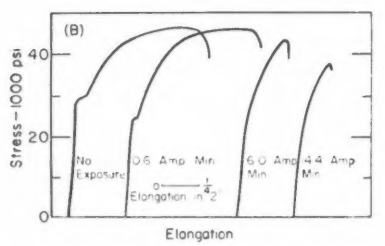


Figure 8—Elimination of the yield point in annealed Armco iron by immersion in aqueous hydrogen sulfide, A, and cathodic charging in sulfuric acid at 2 ma/ sq in, B.

possibly exerted by hydrogen in the molecular form, contained in "traps" existing in the iron or traps produced by local strain during the tensile test; local stresses of this kind would add to the applied stress and cause local yielding with a resulting disappearance of the apparent yield point observed at a single stress.

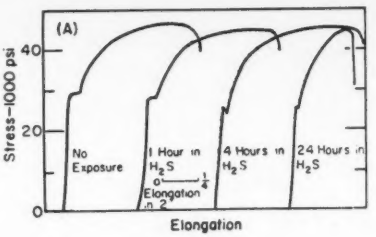
The detailed relation of the observed yield phenomenon to embrittlemen: and fracture is not known now. However, it is clear these observations give a new means of investigating the mechanical effects of hydrogen which, when known in more detail, may provide a means of minimizing generally adverse changes in properties.

Summary of Embrittlement Phenomena

At least with respect to Armco iron, no evidence of phenomena attributable to stress-corrosion cracking was observed. Conversely, the effects of immersion in hydrogen sulfide are consistent generally with effects associated with hydrogen absorption.

Specifically:

1. The time dependence of the decrease in ductility is consistent with a diffusion process.
2. Ductility is recovered in time and the extent of recovery in a given time is dependent on the duration of exposure.
3. Exposure to hydrogen sulfide and cathodic charging in sulfuric acid both result in progressive elimination of the yield point with increasing time of exposure.
4. Stress-strain curves obtained in air after exposure to hydrogen sulfide and those obtained in aqueous hydrogen sulfide differ in a manner consistent with the evolution of hydrogen from steel at atmospheric pressure.



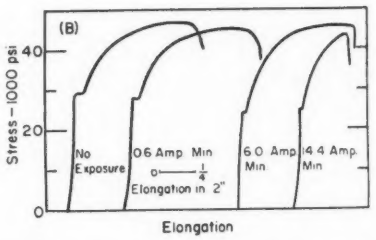


Figure 9—Recovery of yield point elongation in annealed Armco iron after aging 70 days at room temperature following treatment in hydrogen sulfide, A, or cathodic charging, B.

Spontaneous Fracture in Hydrogen Sulfide

The present investigation of fracture phenomena under constant-deformation conditions was a necessary preliminary survey of variables prior to more detailed investigation of specific factors.

Two to six strip specimens, 5 x 1/2 x 1/16 inch were used for each test. A bending stress was applied by the fixture shown in Figure 2; the nominal maximum fibre stress, calculated from the curvature was 140,000 psi. In order to provide a more severe set of conditions, specimens were also bent into a U-shape around a ½ inch mandrel and an elastic stress was superimposed on the plastically deformed specimen by closing the open ends of the deformed specimen.

The criterion of damage was the time to failure. It was observed early in the investigation that failure generally occurred within about one day or did not occur in periods of several hundred hours; accordingly tests were discontinued after a week in the absence of failure.

Quenching Temperature

In order to determine the heat treating time and temperature to be used for iron-nickel alloys in subsequent work, a preliminary investigation of this variable was made. Specimens of 10 percent nickel steel E were heat treated for one hour and quenched to martensite. Results of constant deformation fracture tests in aqueous hydrogen sulfide are given in Table 2.

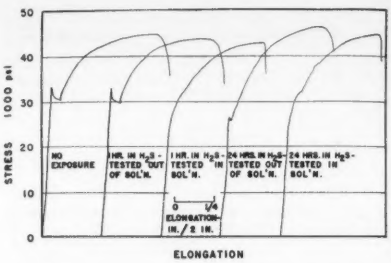

STRESS 1000 psi
NO EXPOSURE
1 HR. IN H_2S - TESTED OUT OF SOL'N.
1 HR. IN H_2S - TESTED IN SOL'N.
24 HRS. IN H_2S - TESTED OUT OF SOL'N.
24 HRS. IN H_2S - TESTED IN SOL'N.
ELONGATION IN./2 IN.
ELONGATION

Figure 10—Comparison of stress-strain curves of annealed Armco iron when tested in and out of a hydrogen sulfide environment.

TABLE 2—Time to Fracture in Hydrogen Sulfide

For 10 Percent Nickel-Iron Martensite

Quenching Temperature (In Degrees C)	Hardness R_c	Time to Failure (Hours)	
		Beam	U-Bend
700	34	2–4	4–7
850	35	3–5	4–7
1000	28	4–11	2
1100	27	2	2
1200	24	7–20	—

Evidently there is no significant difference in the fracture behavior attributable to quenching temperature. For subsequent work the quenching temperature was standardized at 1000C and the time at one hour.

Fracture Susceptibility With Respect To Structure and Composition

The general pattern of fracture behavior in hydrogen sulfide was surveyed for all structures and compositions of materials listed in Table 1 and the results are given in Table 3. Study of these data indicates some interesting correlations among properties, structure and susceptibility to failure.

Mechanical Properties

Neither the 30 percent nickel austenite G nor the 10 percent nickel ferrite E failed under any conditions of constant deformation testing. However, the mechanical properties of both these materials are low and the elastic energy stored in the bent specimens is correspondingly small. In order to evaluate the effect of mechanical properties, the 10 percent nickel ferrite was cold rolled, 66 percent reduction in thickness to the standard 1/16 inch section and its hardness raised thereby to R_c-24. In this condition the material failed in the more severe test. However, cold rolling the 10 percent nickel martensitic structure did not increase its susceptibility to failure and though the difference may not be significant, it appeared to diminish susceptibility to fracture.

Effect of tempering martensite had the anticipated effect of lowering mechanical properties and, if carried far enough, prevented failure.

It was impossible to bring all structures to the same hardness so a direct comparison of susceptibility under the same mechanical conditions could be made. However, it appears that failure of cold worked ferrite is at least indica-

tive of the fact that ferrite is not immune to failure. Addition of 0.44 percent titanium to the 10 percent nickel alloy raises the mechanical properties of ferrite into the range where fracture occurs in constant deformation tests and, as shown in Table 3, causes failure. This result indicates, again, that both martensite and ferrite are susceptible under comparable conditions. Furthermore, the experiment indicates that the addition of an alloying element which forms a stable hydride does not offer a simple solution for fracture caused by hydrogen absorption.

Retained Austenite

X-ray analysis of the 10 percent nickel martensite had demonstrated that retained austenite is absent, within the precision of measurement, ± 0.1 percent. Since this material failed consistently under all conditions, it appears that transformation of retained austenite is not a necessary condition for failure. Because the properties of the 30 percent nickel austenite could not be raised into the range where the material is susceptible to failure, the absence of failure of austenite is inconclusive with respect to the question of the transformation of austenite causing failure.

Additional evidence was obtained, however, by transforming the 30 percent nickel alloy to martensite at —190C. This structure has the same mechanical properties as the 10 percent nickel martensite but failure was never observed under any conditions of stress or deformation in hydrogen sulfide. Again, since the structure contains 5-10 percent retained austenite, it appears that failure in hydrogen sulfide is not necessarily associated with retained austenite or its transformation under stress.

Composition

With respect to nickel concentration in a martensitic structure, both 5 and 10 percent nickel alloys appear to have the same susceptibility to failure. On the other hand, 30 percent nickel martensite does not fail when exposed to hydrogen sulfide even though the mechanical properties are in the range where failure may occur. Later investigation of the hydrogen concentration indicated that this resistance to failure is associated with low hydrogen absorption by 30 percent nickel alloy from hydrogen sulfide, as a consequence of its low corrosion rate.

The effect of oxygen, carbon and nitrogen was investigated in several ways. Specimens of the 10 percent nickel alloy E were decarburized in wet hydrogen until the disappearance of the yield point in accompanying Armco iron specimens indicated a carbon content less than 0.002 percent. Specimens then were cooled slowly in vacuum to remove hydrogen and subsequently reheat treated in argon and quenched to martensite. Fracture tests of the decarburized and nondecarburized specimens showed no significant difference in that both sets of specimens failed in 2.5 hours.

It was anticipated that in the alloy containing 0.44 percent titanium N, the formation of the very stable titanium carbide, nitride and oxide would effectively remove oxygen, nitrogen and carbon. As shown in Table 3, both martensitic and ferritic structures fail in hydrogen sulfide. Provided residual titanium itself does not cause failure, it may be concluded these impurities are not a primary cause of failure.

TABLE 3—Effect of Structure and Composition on Cracking in Hydrogen Sulfide

Material Percent	Mark	Structure	Treatment	R_c Hardness	Failure Time (Hrs) Beam	Failure Time (Hrs) U-Bend
5 Ni	B	Martensite	1000 C quench	27	5–16	4–16
10 Ni	E	Martensite	1000 C quench	28	4–11	2
10 Ni	E	Martensite	1000 C quench, cool to —190 C	27	5–14	5–14
10 Ni	E	Martensite	1000 C quench—Temper 2 hours at 250 C	28	10–20	6–20
			at 400 C	21	4–8	8–10
			(ferrite) at 550 C	11	N.F.	N.F.
10 Ni	E	Martensite	Cold worked 50% reduction	32	13–45	10–20
10 Ni	E	Ferrite	Furnace cooled from 1000 C	10	N.F.	N.F.
10 Ni	E	Ferrite	Cold worked 66% reduction	24	N.F.	9–12
10 Ni-Ti	N	Martensite	1000 C quench	25	3	3
10 Ni-Ti	N	Ferrite	1000 C furnace cool	23	40	6–27
30 Ni	G	Austenite	1000 C quench	R_B58	N.F.	N.F.
30 Ni	G	Martensite	1000 C quench cool to —190 C	28	N.F.	N.F. (21 days)
0.25 C	K	Martensite	920 C brine quench	45	0.5	—
0.37 C	L	Martensite	890 C brine quench	55	0.1	—

TABLE 4—Cracking Susceptibility Under Cathodic Charging

Material Percent	Electrolyte	Structure	R_cH	CS_2+P Poison	Current Density ma/in²	Failure Time (Hrs.) Beam	Failure Time (Hrs.) U-bend	Hydrogen at Saturation cc/100 gm.
10 Ni-E	4 Percent H_2SO_4	Martensite	28	Yes	2	1 N.F. 1 at 7	2	9
10 Ni-E	4 Percent H_2SO_4	Martensite	28	Yes	100–200	N.F.	0.25–0.50	13
10 Ni-E	4 Percent H_2SO_4	Martensite	28	No	100	—	21	3
30 Ni-G	4 Percent H_2SO_4	Austenite	58 (R_B)	Yes	2	—	N.F. in 6 days	9
30 Ni-G	4 Percent H_2SO_4	Martensite	27	Yes	2	—	9–18	30
30 Ni-G	4 Percent H_2SO_4	Martensite	27	Yes	100	—	10–23	—
10 Ni-E	10 Percent NaOH	Martensite	30	Yes	100	—	N.F.	2.5
10 Ni-E	10 Percent NaOH	Martensite	30	No	100	—	N.F.	2.5
0.37 C-L	10 Percent NaOH	Martensite	55	No	No current	N.F. (10 min.)	—	—
Same Specimen	10 Percent NaOH	Current turned on	55	No	100	1 min.	—	—
0.37 C-L	10 Percent NaOH	Martensite	55	Yes	No current	N.F. (22 min.)	—	—
Same Specimen	10 Percent NaOH	Current turned on	55	Yes	100	1 min.	—	—

Induction Period Preceding Fracture

To get information regarding hydrogen as a factor in spontaneous fracture, a comparison was made between fracture time for presaturated and unsaturated 10 percent nickel martensite. One set of six specimens was immersed in hydrogen sulfide without applied stress for 24 hours, which (as determined by hydrogen analysis to be described later) is sufficient to saturate the specimens with hydrogen. After immersion, stress was applied, essentially without removing the specimens from the environment. Failure occurred in an average of 1.9 hours. An identical set of specimens, stressed to the same value before immersion failed in an average time of 4.8 hours.

Interpreting results of the preceding experiment in terms of a stress-corrosion cracking mechanism, postulates corrosion penteration during the period of immersion without stress and a resulting shorter time under stress before failure. However, no evidence of such penetration has been found during much longer periods of exposure. Conversely, the delay time for fracture following saturation with hydrogen was shown by subsequent experiments to be characteristic of the process of fracture associated with hydrogen.

Coated Specimens

Fraser and Treseder[5] conducted a significant set of experiments to distinguish between a hydrogen mechanism and a stress corrosion cracking mechanism. When a narrow band on the tensile side of a bent specimen, opposite the central point of loading, was covered with a vinyl plastic, failure occurred outside the coated region. When the entire tension surface was coated, no failure occurred. When a narrow gap was left in the otherwise complete coating on the tensile surface, failure immediately occurred at the gap in the coating. It was concluded from these experiments that hydrogen was not the primary cause of failure. In drawing this conclusion it was assumed that the partially coated specimens would be saturated with hydrogen and, consequently, they should be as embrittled as specimens without a coating. Because these experiments are very significant with respect to the mechanism of failure, it was

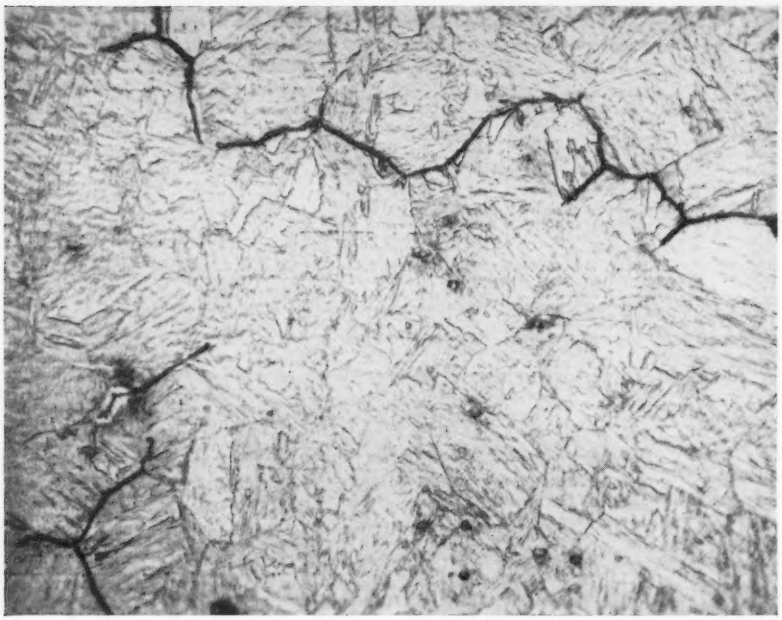


Figure 11—Ten percent Ni E martensite wire. Specimen was cathodically charged with hydrogen unstressed in 4 percent sulfuric acid plus poison at 2 ma/ sq in for 24 hours. Stress of 10,000 psi was then applied and there was no failure in 110 hours. Specimen failed in 15 minutes after stress was raised to 15,000 psi. Cracks were scattered along surface and throughout body of wire. Applied stress direction in horizontal plane. Typical intergranular cracking occurring for this quenched material for both cathodic charging and hydrogen sulfide exposure which follows traces of previous austenitic grain boundaries with directions determined more by internal stress patterns than applied stress. 600X, nital etch.

Figure 12—Ten percent Ni plus 0.44 percent Ti N martensite. Specimen was stressed as U-bend in hydrogen sulfide and failed in three hours. There was intergranular cracking. 900X, nital etch.

important to repeat this work and to reconsider the interpretation.

Specimens of 10 percent nickel martensite and 0.37 percent carbon martensite were used to evaluate the effect of coatings. Three different coatings were employed: an epoxy resin, paraffin and a rubber base material. One set of specimens was coated completely on the tension side and another set was similarly coated except for a gap at approximately half the distance between the center point of the beam and one of the supported ends. The 10 percent nickel specimens did not fail under either of these conditions. The carbon-martensite specimens failed in several hours when completely coated and in a shorter time when a gap was left in the coating. Failure in the latter specimens, however, was not at the gap in the coating but at a nearby location of higher stress.

Interpretation of these experiments appears to be clear. When hydrogen is generated at one side of the specimen, a concentration gradient in hydrogen exists across the specimen from the exposed to the coated side; in effect the latter becomes an "outer" surface. When susceptibility to failure is high, the concentration near the coated, tensile surface may be sufficiently great to cause failure. In materials of lower susceptibility, the necessary concentration at the tensile surface is not attained. Because subsequent experiments have shown that both stress and hydrogen concentration are involved, failure of carbon martensite at a location of higher stress in the vicinity of a gap in the coating is not unexpected.

If it is assumed that a linear concentration gradient exits in a specimen coated on one side, the average hydrogen content should be one-half of that found in a saturated specimen exposed on both sides. A direct test of this assumption was made by determining the hydrogen content of 10 percent nickel martensite specimens, each coated on one side with one of the three coating materials and immersed in hydrogen sulfide for 50 hours. Average hydrogen content of four specimens was 1.8 ml/100 gm. The saturation concentration of hydrogen in specimens exposed on both sides, under the same chemical conditions, is 3.5 ml/100 gm. Agreement with the assumed linear gradient is satisfactory.

It appears, therefore, that evidence from experiments with coated specimens does not exclude hydrogen as a primary cause of failure. The experiments only emphasize that it is necessary to have a sufficient concentration of hydrogen, above some limiting value which depends on the type of steel and its properties at a location of tensile stress.

Failure Under Conditions of Cathodic Charging

As a result of the preceding work, it is obviously necessary to evaluate the effect of hydrogen introduced with a minimum of corrosion. For this purpose specimens were charged cathodically in 4 percent sulfuric acid and in 10 percent sodium hydroxide, with and without carbon disulfide as a cathodic "poison." Stress was applied by bending in the same manner as in previous, constant deflection experiments. The results of these tests are given in Table 4, together with the approximate hydrogen concentration at saturation. In correlating hydrogen content with failure, it should be remembered that the U-bend specimens are plastically deformed to an uncertain extent and that this deformation greatly increases the hydrogen content.

In common with others who have investigated spontaneous cracking under these conditions, reproducibility and consistency of results is not good. However, the general pattern is reasonably clear. The 10 percent nickel martensite fails rapidly and consistently under the more severe conditions of plastically deformed specimens. Furthermore, the carbon disulfide poison is not a necessary condition of failure. This material,

charged in sodium hydroxide, did not fail but, as indicated, the hydrogen concentration absorbed under these conditions is lower than that absorbed from sulfuric acid containing carbon disulfide and slightly lower than the concentration resulting from charging without the sulfide poison.

In previous work, it was demonstrated that 30 percent nickel martensite specimens did not fail in hydrogen sulfide under any conditions of stress. Under the present conditions of cathodic charging, this material failed consistently and later analysis for hydrogen gave the apparent reason when it was found that the saturation concentration of hydrogen under these conditions was one of the highest observed in any material except Armco iron and about 50 times that absorbed from hydrogen sulfide.

To show conclusively that fracture could occur solely due to the action of hydrogen and that fracture was not dependent on a corrosion reaction, the most susceptible carbon martensite was immersed in sodium hydroxide while under stress but without applied cathodic current. Failure never occurred under these conditions. However, failure invariably occurred, almost immediately, when a cathodic current was applied to the specimen.

Structural Path of Fracture

Fraser and Treseder[5] have demonstrated that failure in hydrogen sulfide generally is associated with a single macroscopic crack, instead of the numerous branching cracks characteristic of stress corrosion cracking. This distinctive failure process was confirmed in the present work and, in fact, so few subsidiary cracks were observed that microscopic identification of the structural path of fracture was difficult.

In the 10 percent nickel martensite, the failure path in hydrogen sulfide, and as a result of cathodic charging, follows the traces of original austenite grain boundaries. Micrographs of typical failure patterns are shown in Figures 11 and 12 for cathodic charging conditions and exposure in hydrogen sulfide, respectively. The general appearance of these structures is identical with those observed by Prange[5] in commercial 9 percent nickel steel.

The 30 percent nickel martensite also failed at austenite boundaries, as far as could be ascertained. The number of subsidiary cracks was invariably small and it was difficult to resolve structural details because of the resistance to etching. Figure 13 illustrates a few subsidiary cracks which are certainly intergranular with respect to the prior austenite boundaries.

The fracture path in 10 percent nickel ferrite, studied under dead-loading conditions, could not be clearly identified. Figure 14 shows the single macroscopic fracture which occurred in 54 hours in a wire stressed to 45,000 psi and charged with hydrogen at 100 ma/in.^{2} Apparent curvature is caused by misalignment in the mounting of the two parts of the fractured specimen. The complete brittleness of the failure is evident; the small volume of plastically deformed material at one edge of the fracture path is caused by tensile failure of the section ahead of the advancing crack. Detailed examination of the structure indicated a transgranular fracture with respect to the ferrite phase. However, ferrite grain boundaries are characteristically irregular and interpretation may not be complete. Furthermore, original traces of austenite boundaries are swept by growing ferrite grains and are no longer visible. Examination of many structures did not reveal any significant feature which could be identified as defining the fracture path.

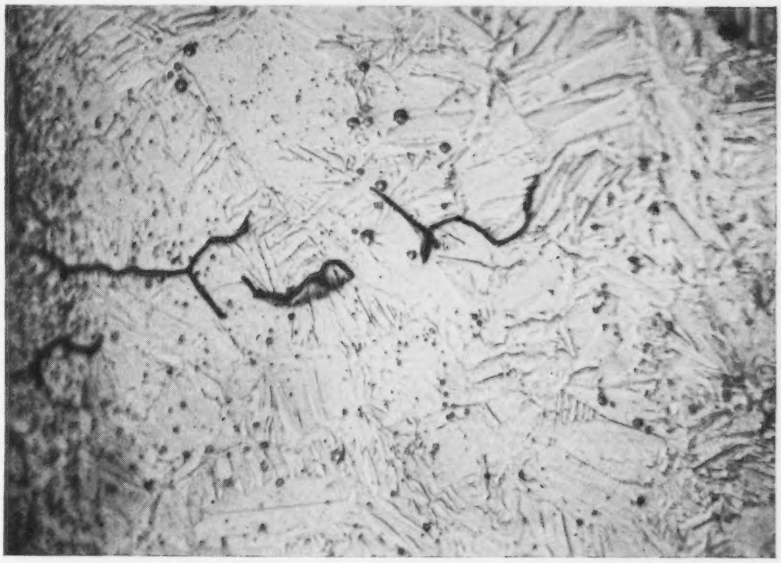


Figure 13—Martensite in 30 percent Ni G material obtained by cooling to —190 C in liquid air after quenching from 1000 C R$_c$H 28. Specimen was stressed as U-bend and cathodically charged with hydrogen at 100 ma/ sq in with poison. Sample failed in 12 hours. Applied stress was vertical. Note intergranular cracking typical of martensites. Sample was etched with 3:2 nitric-acetic acids. 600X.

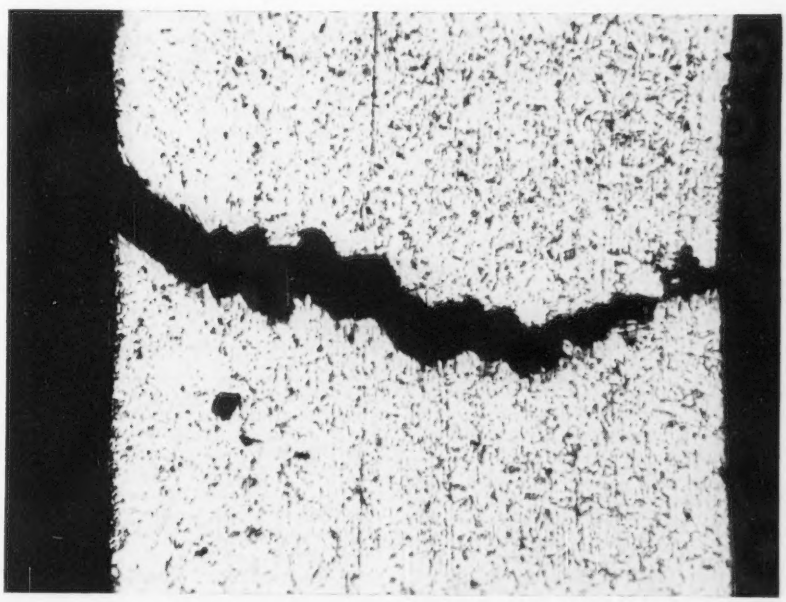


Figure 14—Ten percent Ni E ferrite wire cathodically charged unstressed for 24 hours with poison at 100 ma/ sq in. Stress of 45,000 psi was applied and sample failed in 54 hours. Main fracture is pictured. Photograph shows predominant brittleness of fracture with but a slight evidence of ductility at the far left. This probably was due to the high stresses created at this point by the reduction in cross-sectional area caused by the advancing crack. 100X.

Structural evidence with respect to the path of fracture was obtained under conditions in which the time of exposure was relatively short. To evaluate susceptibility to intergranular corrosion, which usually is apparent after prolonged immersion in the environment in which stress corrosion cracking is observed, specimens of all alloys and structures were immersed without applied stress in hydrogen sulfide for 35 days. Subsequent examination showed no structural attack indicative of stress corrosion cracking susceptibility. Figure 15 shows

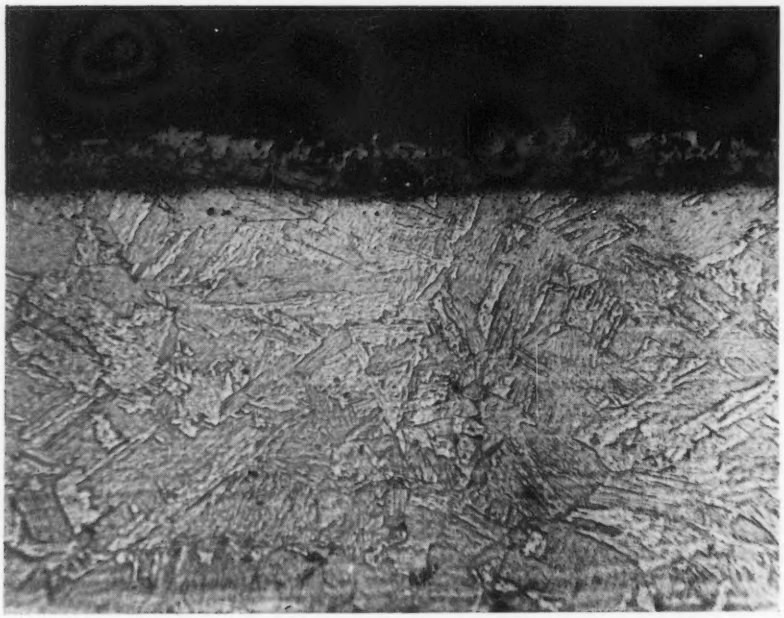


Figure 15—Ten percent Ni E martensite. Sample was exposed unstressed 35 days in hydrogen sulfide to detect any corrosion penetration of the surface. Specimen bent slightly to open any possible cracks. Tensile surface. FeS corrosion product is on surface. There was a similar lack of surface penetration for all materials given this same test. 250X.

a representative cross section of the surface of a 10 percent nickel martensite specimen, one of the structures most susceptible to failure in hydrogen sulfide, which has been bent slightly to open incipient cracks. There is no structural attack.

Metallographic observations in the present work appear to disagree with those of others[8] who have found surface pitting in hydrogen sulfide, accompanied by failure originating at the pit. But, it is clear that if a pit is present, the probability of failure at this point is high; on the other hand, failures not associated with pits have been observed in the present experiments which appear to indicate that fracture is not necessarily associated with the prior formation of a pit.

Summary of Embrittlement and Fracture Phenomena

1. Immersion of iron and alloy steels in hydrogen sulfide produces all the familiar "embrittling" effects associated with the absorption of hydrogen by steel; recovery of properties in time from the effects of immersion indicates that hydrogen rather than corrosion damage is responsible for the loss of mechanical properties.

2. The introduction of hydrogen by cathodic charging and immersion in hydrogen sulfide cause the suppression of the yield point in Armco iron. This new phenomenon subsequently has been verified and extensively studied by others. Again, the effect disappears in time and evidently is not related to corrosion damage.

3. Microstructural observations do not indicate any particular structural phase as a cause of failure or a preferred corrosion path.

4. Failure in constant deformation tests occurs in completely ferritic or martensitic structures but failure does not occur in hydrogen sulfide in structures containing martensite and 5-10 percent retained austenite (30 percent nickel-iron). Thus, neither martensite nor retained austenite appear to be a necessary condition of failure.

5. Failure by cathodic charging in acid and alkaline solutions indicates that corrosion is not a necessary prerequisite for failure. The fact that at least one alloy (30 percent nickel martensite) does not fail in hydrogen sulfide, in which the quantity of hydrogen absorbed is small and it does fail when the hydrogen content is raised by cathodic charging, strongly indicates that failure is closely associated with hydrogen concentration.

6. The observed dependence of failure, in a constant deformation test, on mechanical properties indicates that a lower limit of stress may exist below which failure does not occur for a given hydrogen concentration.

7. No correlation between failure and the presence or absence of traces of oxygen, carbon or nitrogen has been observed.

8. Finally, failure occurs under conditions in which no structural corrosion has been observed after prolonged immersion in hydrogen sulfide. While the absence of apparent, structurally defined, corrosion does not absolutely exclude stress corrosion cracking as the primary mechanism, it does indicate that another mechanism may be responsible for failure.

Within the limitations of present data, it appears that the primary cause of failure is associated with hydrogen absorbed from hydrogen sulfide. To substantiate this conclusion, it is necessary to determine whether it also is consistent with a quantitative determination of the amount of hydrogen absorbed by the different alloys. The following section of this report will be devoted to measurement of hydrogen absorption in the austenitic, martensitic and ferritic iron-nickel alloys used in the preceding experiments.

Part 2—Hydrogen Absorption in Iron-Carbon and Iron-Nickel Alloys

Previous embrittlement and constant-deformation fracture experiments (Part 1) indicated that spontaneous failure of iron-nickel and iron-carbon alloys in hydrogen sulfide is associated with the absorption of hydrogen. The Part 2 study was undertaken to determine whether the proposed cause of failure is consistent with a quantitative analysis of hydrogen absorption and permeation in the same alloys and structures used in the previous fracture experiments.

The most recent bibliography concerned with hydrogen embrittlement[17] contains 1200 references. However, in only a negligible fraction of the work represented in this compilation have observed changes in mechanical properties been combined with a quantitative measurement of hydrogen content. So, available data are not generally useful because composition of the alloy in hydrogen is unknown and extremely variable. Furthermore, although hydrogen usually is absorbed under conditions of effective high pressure, testing is conducted most frequently at atmospheric pressure with the result that hydrogen content changes in time, preceding and during the test. It is not surprising, therefore, that available data are not quantitatively useful and often are contradictory.

The present problem is concerned with the absorption of hydrogen by iron alloys from aqueous solutions at room temperature, either as a result of corrosion or the reduction of hydrogen ions by a current from an external battery. In these experiments no hydrogen gas pressure above atmospheric is developed on the specimen surface. To use thermodynamic relationships it is necessary to use the "fugacity" concept which may be regarded as the virtual pressure of an ideal gas. For hydrogen, fugacity may be considerably greater than the pressure and to increase with pressure.

Assuming that the observed electrical potential of steel in an aqueous solution, relative to the hydrogen electrode in the same solution, is a measure of hydrogen overvoltage, the potential and the fugacity are related by:

$$E = \frac{2.3\ RT \log f_{H_2}^{1/2}}{F}$$

where E is the potential in volts and f is the fugacity in atmospheres. Hydrogen overvoltages on iron at low current densities are of the order of 0.2 volts and, accordingly, the apparent fugacity is approximately 10^6 atmospheres. Irrespective of how the fugacity is calculated, it is always astonishingly high and in the ranges of 10^6-10^8 atmospheres.[18]

The concentration of hydrogen in a solid metal may be related approximately to the pressure and temperature through the equilibrium constant applying to the two phases. Assuming an ideal gas and an ideal solid solution:

$$\tfrac{1}{2}\ H_2\ (\text{gas}) = H\ (\text{in metal})$$

$$K = \frac{H}{P_{H_2}^{1/2}}$$

$$\text{Log}\,[H] = \frac{-\Delta H^\circ}{2.3\,RT} + \frac{\Delta S^\circ}{2.3\,R} + \frac{1}{2}\log P_{H_2}$$

where ΔH° and ΔS° are the change in enthalpy and entropy, respectively, accompanying the solution of ½ mole of hydrogen in iron. Values for ΔH° and ΔS°, deduced from high temperature solubility data for alpha and gamma iron by Geller and Sun,[19] are as follows.

Alpha Iron:
$\Delta H = 6500$ cal/0.5 Mole H_2
$\Delta S = 7.7$ cal/deg/0.5 mole H_2

Gamma Iron:
$\Delta H = 5400$ cal/0.5 mole H_2
$\Delta S = 7.7$ cal/deg/0.5 mole H_2.

The smaller value of enthalpy for gamma iron indicates a higher solubility of hydrogen in gamma iron which is, indeed, observed at one atmosphere pressure.

The preceding equilibrium constant indicates that solubility is proportional to the square root of pressure at constant temperature. While this pressure dependence is observed at pressures below 100 atmospheres there is no certainty this relationship will be observed in the range of pressures of interest in the present work. Accordingly, extrapolation of isobaric data, mostly obtained at high temperatures and one atmosphere pressure, to room temperature generally is precluded. In any case, it has been shown that the overvoltage and fugacity are exponentially related and, consequently, the effective pressure in an aqueous system at room temperature is extremely sensitive to changes in overvoltage, which also is dependent on minute traces of surface-active impurities.

In summary, with respect to hydrogen absorption at room temperature, extrapolations of available data to conditions prevailing in an aqueous system at room temperature and very high effective pressures probably are not justifiable. Also, considerable uncertainty exists as to the magnitude of the effective pressure and the factors controlling the pressure. The latter uncertainty is responsible for the apparently conflicting results which frequently are observed, particularly when an effect of hydrogen is measured without corresponding data on the quantity of hydrogen actually absorbed by the material.

Absorption at room temperature involves another type of problem which generally is not given adequate consideration. The diffusion rate of hydrogen into iron is high, but a significant period of time is required to saturate a specimen of reasonable size at room temperature. Thus, in order to establish that equilibrium has been attained and that no concentration gradient prevails in the specimen, it is necesary to determine the time dependence of absorption. Furthermore, it is necessary also to saturate a specimen before mechanical tests, otherwise the conditions are not well defined and, again, considerable uncertainty is introduced regarding hydrogen concentration and its distribution relative to the outer surface.

In the following work, the quantity of hydrogen absorbed by the alloys and structures used in investigating spontaneous fracturing has been determined. Because effective pressure resulting from a corrosion reaction in aqueous hydrogen sulfide depends on corrosion rate and, therefore, on composition, it was necessary also to determine the quantity of hydrogen absorbed by cathodic charging under conditions of constant current density. Under the latter conditions the effect of composition may be separated from the effect of structure and composition in the various alloys required to obtain austenite, martensite and ferrite.

Experimental Procedure

Materials and Treatment

The same alloys were used for hydrogen solubility determinations as were used in Part 1 for studying fracture susceptibility. In particular, the series of iron-nickel alloys provided a means of comparing hydrogen absorption in ferrite and martensite directly, in an alloy of the same composition (10 percent Ni) and austenite and martensite in another alloy (30 percent Ni). Armco iron and iron carbon alloys also were used to determine the specific effects of composition. The necessary heat treatments all were conducted in purified argon. Specimens were sheets ½ x 1/16 x 5 inches long.

Apparatus and Procedure

For the purpose of determining hydrogen content, hydrogen was collected over mercury at 200 C. Darken and Smith[18] found that extraction at 170 C yielded 90 percent of the hydrogen that could be extracted in vacuum at 800 C and that the gas evolved contained 95 percent hydrogen. For the present purpose, this method also had the advantage of simplicity which made it possible to make a large number of determinations and comparisons; furthermore, previous work indicated that mechanical properties were recoverable in a short time at 100 C and it was assumed that the hydrogen extracted at 200 C represented the fraction of hydrogen responsible for the change in properties.

A standardized procedure was adopted. Specimens exposed in aqueous hydrogen sulfide or to cathodic charging were rinsed in water and in three successive beakers of acetone, dried in an air blast and inserted in a mercury filled tube. The time required to transfer the specimens from solution to the mercury tubes was 2-4 minutes.

The tube was closed at one end with a calibrated centrifuge tube below which was a constriction preventing the specimen from rising above the mercury. The mercury filled tube containing the specimen was inserted into a vertical tube furnace about twice as long as the specimen. A battery of eight such furnaces was supported on a rack for simultaneous determinations. The specimens were heated to 200 C for 6-12 hours after which the assembly was cooled to room temperature. The volume of mercury displaced by hydrogen was measured and suitable corrections made to reduce all readings to standard conditions of 0 C and 760 mm of mercury pressure.

Runs made with unexposed specimens showed that blank corrections were negligible. The period of six hours at 200 C was determined as being more than sufficient to extract all available hydrogen. One source of error, common to all experiments, was the loss of hydrogen during transfer from solutions to analysis tubes. Figure 16 shows the rate of evolution of hydrogen at room temperature from 10 percent nickel-iron martensite after 24 hours' exposure in aqueous hydrogen sulfide. Loss of hydrogen in transfer is not greater than 5 percent and, judging from the equality of the data for the three specimens shown in Figure 16, the error is constant. This error is within the general level of reproducibility and, accordingly, a correction was not applied to the results.

Duflot[20] reported that reproducibility was much improved if specimens were electropolished prior to cathodic charging. However, four specimens cathodically charged at 2 ma/in.^{2} for 25 hours after electropolishing in a perchloric acid-acetic acid electrolyte yielded 39.5, 42.6, 69.5, and 72.7 ml of hydrogen per 100 grams of iron, an average deviation from the mean of 26 percent. Generally, deviations were not as large as this and it was concluded that electropolishing was not warranted. Accordingly, all specimens were used as they came from the heat treating operation, without further surface preparation.

Exposure conditions were identical to those used in previous fracture experiments and involved immersion in saturated, aqueous hydrogen sulfide and cathodic charging in 4 percent sulfuric acid. In the latter experiments, a surface "poison" consisting of a few drops per liter of carbon disulfide containing phosphorus was employed generally. Except when it was the subject of investigation, the current density was maintained constant at 2 ma/in.^{2}

Treatment of Data

As indicated by experiment, initial concentration of hydrogen in steels used in these experiments is negligible in comparison with concentrations determined after immersion in hydrogen sulfide or after cathodic charging. Assuming that the surface reaches equilibrium immediately with respect to hydrogen at an effective (high) pressure, which is defined by the current density, the average concentration, C_m, at any time inside the steel will depend on the time and the rate of diffusion. For constant boundary conditions of pressure and dimensions of the specimen, it may be assumed also that there is a constant saturation concentration, C_s, which is attained throughout the specimen at some time and which is reached at the surface immediately after immersion.

If the diffusivity, D, is constant, it can be shown[21] that the total amount of substance diffusing across unit area into a semi-infinite medium from $t = 0$ to t is proportional to $\sqrt{Dt} \cdot C_s$. Consequently the concentration of hydrogen in specimens, exposed to absorption conditions for periods of time short of the time required for saturation, should be proportional to the square root of exposure time. The data indicate that this relationship is obeyed and, therefore, all results are plotted as a function of $t^{1/2}$.

With the exception of one special case, a saturation concentration was observed. In many experiments the linear absorption rate with respect to $t^{1/2}$ extends significantly beyond the ultimate saturation limit; while this phenomenon is observed most frequently as a result of cathodic charging, it also is found in several instances after immersion in hydrogen sulfide. The data indicate "supersaturation" which has not been explained adequately.

The numerous variables studied may be organized in several ways. In the fol-

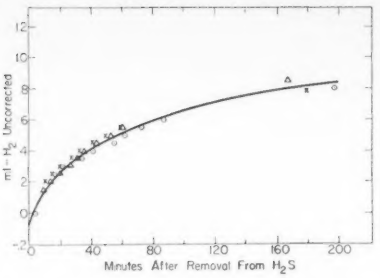


Figure 16—Evolution of hydrogen at room temperature from three specimens of 10 percent Ni-Fe martensite, after 24 hours in aqueous hydrogen sulfide. Readings are uncorrected to STP; total volume of hydrogen, at the same pressure, extracted at 200 C is approximately 1.8 ml.

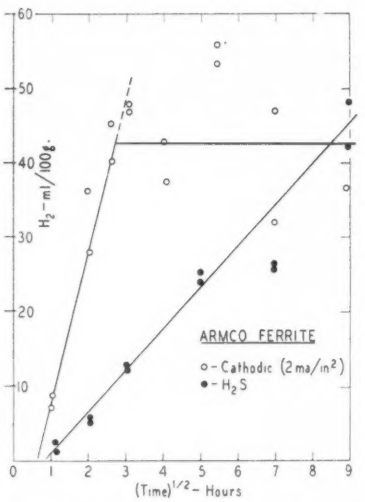


Figure 17—Hydrogen absorbed by annealed Armco ferrite from hydrogen sulfide or cathodic charging.

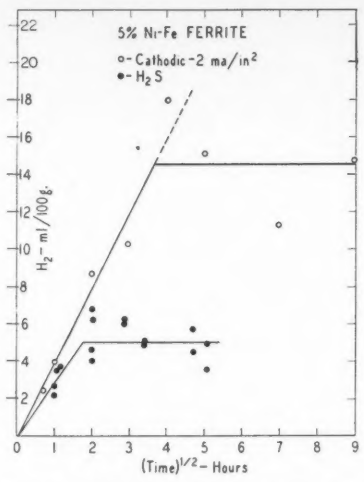


Figure 18—Hydrogen absorbed by 5 percent Ni-Fe ferrite from hydrogen sulfide or cathodic charging.

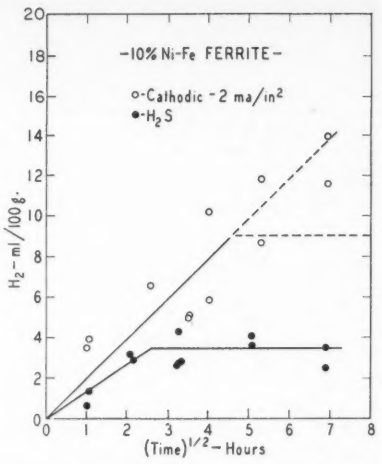


Figure 19—Hydrogen absorbed by 10 percent Ni-Fe ferrite from hydrogen sulfide or cathodic charging.

lowing presentation of data, hydrogen absorbed from hydrogen sulfide and from cathodic charging at 2 ma/in.² generally are presented together arranged in terms of composition, structure, plastic deformation and chemical variables of which the effect of sulfide ion, current density and alkaline electrolytes are considered in detail.

Experimental Results

Structure and Composition

Figures 17, 18 and 19 show the quantity of hydrogen absorbed from hydrogen sulfide and from cathodic charging at 2 ma/in.² by ferritic structures containing 0 to 10 percent nickel.

Of major interest is the anomalous behavior of Armco iron in which the hydrogen concentration does not appear to reach a saturation limit in hydrogen sulfide. The limit is not well defined under cathodic charging conditions but it appears to be real. Present interpretation associates the extremely large concentrations observed in Armco iron with blistering in this material, as a consequence of which hydrogen may accumulate in the growing blisters more or less without limit.

Decreasing saturation concentration observed in hydrogen sulfide with increasing nickel content may be ascribed most simply to the decreasing corrosion rate. However, while the data are not quite adequate for absolute proof, it appears that a net decrease in hydrogen content at saturation may be deduced from Figures 18 and 19 when cathodic charging at constant current density is employed.

Figure 20 shows hydrogen absorption in a 10 percent nickel martensite structure of the same composition as material used in preceding measurements of absorption in ferrite.

First, with respect to absorption from hydrogen sulfide, there is ample evidence showing that absorption is proportional to the square root of exposure time, up to a limiting value. Saturation concentration is well defined and is clearly constant in time to 200 hours.

Second, comparison with the data for ferrite in Figure 19 shows that the rate of absorption and the amount of hydrogen absorbed by ferrite is identical with that absorbed by martensite. Evidently, in this case, the composition (and corrosion rate) is much more significant than the structure in defining the hydrogen concentration.

Third, comparison of the data for ferrite and martensite, under conditions of constant current density, indicates that the saturation concentration is the same; but, the rate of absorption is greater for martensite than ferrite.

Figure 21 presents the data for 30 percent nickel austenite and martensite. It is again evident that increasing the nickel concentration greatly decreases the saturation concentration absorbed from hydrogen sulfide and that conversion of the structure to martensite of the same composition does not affect the concentration of hydrogen absorbed from hydrogen sulfide.

Under conditions of cathodic charging at constant current density (effective pressure), it is interesting to note that the saturation concentration in 30 percent nickel austenite is apparently the same as in 10 percent nickel martensite and ferrite. However, conversion of 30 percent nickel austenite to martensite of the same composition produces a very large change in the saturation concentration and in the absorption rate.

In order to evaluate the effect of carbon content, two iron-carbon alloys containing 0.25 and 0.37 percent carbon were made specially for this work from Armco iron. Figure 22 showing the results for immersion of 0.37 percent C ferrite in hydrogen sulfide and cathodic charging at 2 ma/in.² indicates that there is little or no difference in the rate of absorption or in the concentration of hydrogen absorbed by the two methods. The equality of the saturation limit, in this case, appears to be the result of a coincidence in effective current density and probably does not have any other particular significance.

Figure 23 shows the results of absorption from hydrogen sulfide by carbon martensite of two different carbon concentrations. Evidently, the carbon content, and tetragonality of the structure and associated internal stress, have no significant effect on the saturation concentration of hydrogen.

While the concentration of hydrogen in iron carbon alloys appears to be independent of carbon content, within the indicated composition limits, there are significant details which do not appear in Figures 22 and 23. It was impossible to charge carbon martensite cathodically at 2 ma/in.² with a sulfide poison because the material invariably disintegrated. Thus, while the necessary data could not be obtained directly, it appears that the hydrogen content of carbon martensite, absorbed during cathodic charging, must be larger than that absorbed from hydrogen sulfide because disintegration did not occur under the latter conditions. It also is interesting to note that addition of 0.25 percent carbon, in the form of carbide in pearlite, produces a considerable reduction in saturation concentration in Armco iron. This decrease is more than a matter of corrosion rate because the difference is maintained under cathodic charging. Also, while the saturation concentration of hydrogen absorbed by ferrite and carbon martensite from hydrogen sulfide is the same, the rate of absorption by martensite is considerably greater, as indicated by the slope of the curves in Figures 22 and 23.

Figure 24 summarizes the principal results of absorption rate and hydrogen concentration in the various structures and compositions investigated in hydrogen sulfide. It is apparent that composi-

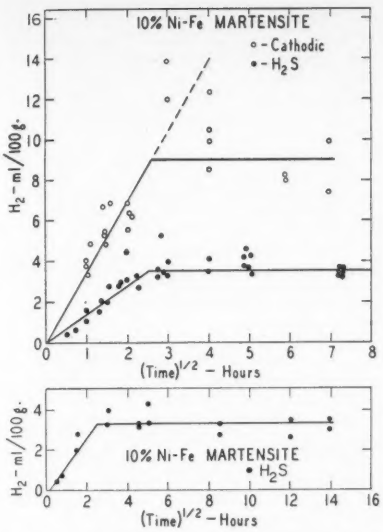


Figure 20—Hydrogen absorbed by 10 percent Ni-Fe martensite from hydrogen sulfide or cathodic charging.

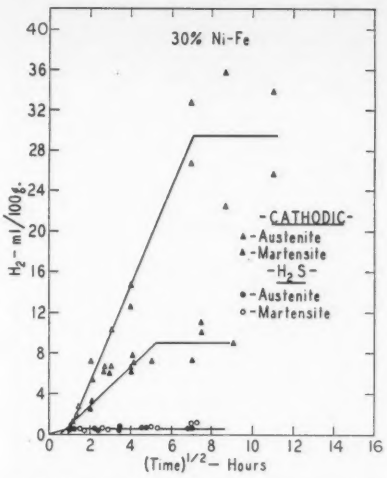


Figure 21—Hydrogen absorbed by 30 percent Ni-Fe austenite and martensite from hydrogen sulfide or cathodic charging.

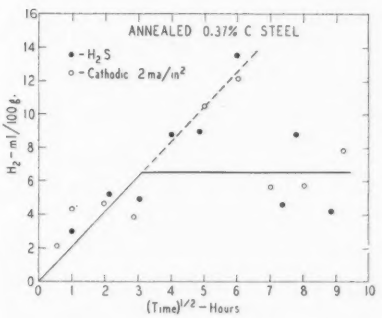


Figure 22—Hydrogen absorbed by annealed carbon steel from hydrogen sulfide or cathodic charging.

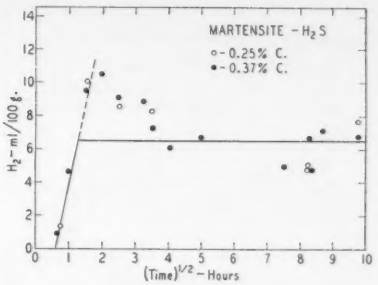


Figure 23—Hydrogen absorbed by different carbon martensites from hydrogen sulfide.

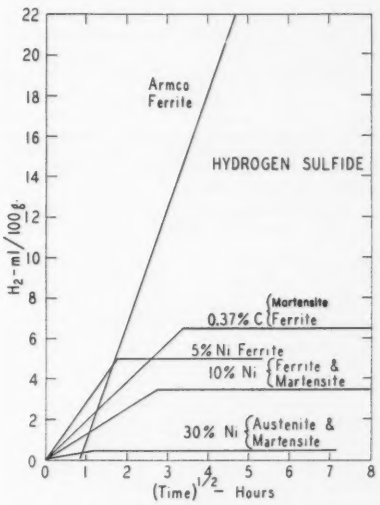


Figure 24—Comparison of hydrogen absorbed from aqueous hydrogen sulfide by different structures and compositions of Ni-Fe alloys.

tion is one of the most significant factors. In particular, nickel and carbon both reduce the saturation concentration. However, this reduction is not proportional to the corrosion rate because the measured reduction in corrosion rate with the addition of 10 percent nickel to Armco iron is approximately 30 percent while the corresponding reduction in hydrogen content is much greater. It is very significant, with respect to the present problem of spontaneous fracture, that the apparent hydrogen content resulting from immersion in hydrogen sulfide is independent of whether the structure is austenitic, martensitic or ferritic, in an alloy of constant composition.

Figure 25 summarizes results of hydrogen absorption from cathodic charging at 2 ma/in.^{2} in sulfuric acid containing carbon disulfide. If hydrogen content were independent of structure and composition and dependent only on effective pressure, all data would fall on a single curve, which is obviously not the case. Absorption rate and saturation concentration appear to be dependent on both composition and structure. Saturation concentration falls rapidly with nickel content and the effect of austenite, in comparison with ferrite, is lost in the compositional dependence. However, transformation of 30 percent nickel austenite to martensite results in a really large increase in concentration and absorption rate.

Plastic Deformation

Earlier work had shown that the ductility of Armco iron decreased linearly with cold reduction as a result of exposure to hydrogen sulfide for 10 hours and that this decrease was a net change due to hydrogen, after correcting for the effect of cold work.

To evaluate the change in the saturation concentration of hydrogen, tests were conducted with 10 percent nickel ferrite. Material, originally 3/16 inch thick, was rolled to different thicknesses such that, after annealing for one hour at 1000 C, subsequent reductions to a common thickness of 1/16 inch resulted in reductions varying in amount up to 66 percent. These specimens were exposed for 25 hours in hydrogen sulfide, more than twice the time required for saturation and the hydrogen content determined. Results shown in Figure 26 indicate that the hydrogen content increases by a factor of 2.5 and subsequently decreases with continued reduction in thickness.

Present results generally agree with previous observations[18, 22] except that a maximum has not been observed previously. More extensive work to prove the reality of this maximum is indicated so it would be best to assume a constant limiting effect of cold work. Duflot[20] observed that absorption in Armco iron first decreased to a minimum and then increased. However, the minimum was observed only when the specimens were electropolished before charging and not otherwise.

The Effect of Carbon Disulfide on Hydrogen Absorption

Significance of the various components of the phosphorus-carbon disulfide "poison" was investigated by charging 10 percent nickel martensite at 2 ma/in.^{2} in 4 percent sulfuric acid. Figure 27 presents results of cathodic charging a) without a poison, b) with the addition of twenty drops of carbon disulfide to 900 ml of 4 percent sulfuric acid and c) with the addition of an equal amount of a solution of 2 grams of yellow phosphorus in 40 ml of carbon disulfide.

Saturation concentration and rate of absorption are not affected by the presence of phosphorus. Effect of carbon disulfide however, is very large, increasing the saturation concentration of hydrogen by a factor of 15 and also greatly increasing the absorption rate. The nature of the corresponding effects of carbon disulfide and the sulfide ion derived from dissociation of hydrogen sulfide is not clear because of the uncertain state of carbon disulfide in dilute sulfuric acid.

Hydrogen Absorption as a Function of Current Density

A primary variable in cathodic charging is current density. Figure 28 shows results of a study of the saturation concentration of hydrogen as a function of current density in sulfuric acid with and without sulfide poison. In both cases, time dependence was determined at each current density to insure that saturation was attained.

Data are in conformity with a relationship of the form:

$$C_s = a + b \log I$$

where C_s is the saturation concentration of hydrogen in ml/100g, I is current density in ma/in.^{2} and a and b are constants. This relationship is, of course, of

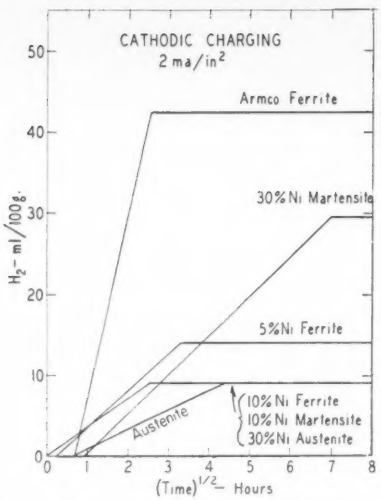


Figure 25—Dependence of hydrogen absorption on structure and composition when cathodically charged in 4 percent sulfuric acid with poison at 2 ma/sq. in.

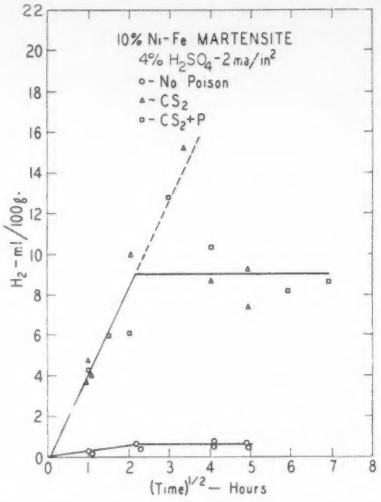


Figure 27—Dependence of hydrogen concentration absorbed by 10 percent Ni-Fe martensite on a trace of carbon disulfide in the electrolyte.

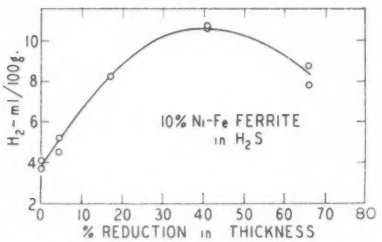


Figure 26—The dependence of hydrogen absorption on plastic deformation.

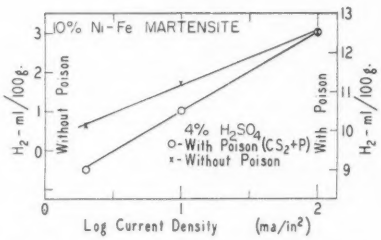


Figure 28—Linear relationship of hydrogen absorption and log current density for cathodic charging of 10 percent Ni-Fe martensite.

the same form as the Tafel equation for the dependence of hydrogen overvoltage on current density.

Hydrogen Absorption from Sodium Hydroxide

Martensitic, 10 percent nickel-iron specimens were cathodically charged with hydrogen in 10 percent sodium hydroxide at current densities of 2 and 100 ma/in.². The results, Figure 29, show little or no systematic arrangement, either with respect to the current density or the effect of the sulfide poison.

It is evident, however, that the general level of hydrogen concentration is lower than that absorbed from hydrogen sulfide and much lower than the amount absorbed from sulfuric acid at the current densities employed in the present experiment.

Summary of the Effect of Structural and Chemical Variables on Hydrogen Absorption

It can be shown[21] that the total amount of diffusing substance per unit area which enters a semi-infinite solid is:

$$2\sqrt{Dt}\,(C_s - C_o)\int_o^\infty (1-\phi)\,\frac{d(X)}{2\sqrt{Dt}}$$

The definite integral has the value of $1/\sqrt{\pi}$ when ϕ is the probability integral. In the present example, C_s is the concentration at saturation and C_o is the initial concentration at $t = 0$, assumed to be zero. Accordingly, the amount of hydrogen crossing unit area in time t is:

$$C = 1.13\sqrt{Dt}\cdot C_s\ (ml/cm^2)$$

Further simplification is possible by taking the time for half saturation, in which case the diffusion constant, D, is inversely proportional to the time for half saturation, $t_{1/2}$; introducing the necessary units, and taking $t_{1/2}$ (hours):

$$D = \frac{3.9 \times 10^{-7}}{t_{1/2}}\ cm^2/sec.$$

Table 5 summarizes experimental data and the apparent diffusion constants, calculated in accordance with the preceding expression, for a set of constant chemical conditions (constant effective pressure and 25 C).

While the present experiments were designed primarily to determine saturation concentrations and not diffusion constants, it appears that diffusion data are obtainable from the non-steady state absorption measurements. Almost all such data previously have been calculated from high temperature concentration and permeation experiments, or directly from permeation experiments at room temperature. In the latter case, D is inversely proportional to the time required to attain steady-state flow through a diaphragm and this time is extremely sensitive to surface conditions at the entrance side of the diaphragm. For iron, the accumulated data[19, 24] from many experiments indicate that D_{298} is approximately 10^{-5} cm²/sec. The data in Table 5, obtained from the present experiments, are from one to two orders of magnitude lower than the previous values.

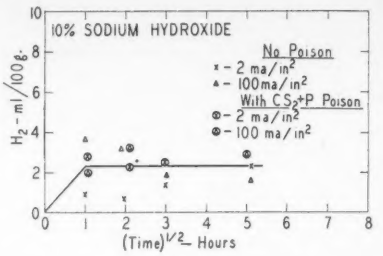


Figure 29—Hydrogen absorbed by 10 percent Ni-Fe martensite when cathodically charged in a sodium hydroxide electrolyte.

TABLE 5—Diffusion Data
Cathodic Charging, 4% H_2SO_4 + CS_2, 2 ma/in.²

Structure	C_s (ml/100g)	t_s (Hours)	$t_{1/2}$ (Hours)	D x 10^7 (cm²/sec)
Armco ferrite	(42.)	(7.4)	(2.0)	1.9
5 Ni ferrite.	14.5	14.0	3.6	1.1
10 Ni ferrite.	9.0	20.	6.3	0.62
10 Ni martensite.	9.0	6.3	1.6	2.4
30 Ni austenite..	9.0	25	7.5	0.52
30 Ni martensite.	30.0	49	14.5	0.28

TABLE 6—Diffusion Data
Absorption of Hydrogen from Hydrogen Sulfide

Structure	C_s (ml/100g)	t_s (Hours)	$t_{1/2s}$ (Hours)	D x 10^7 (cm²/sec)
0.25-0.37C ferrite.....	6.5	9	2.3	1.7
0.25-0.37C martensite.	6.5	1.0	0.32	12.0
5% Ni ferrite.....	5.0	3.1	1.1	4.9
10% Ni ferrite.....	3.5	6.3	1.5	2.5
10% Ni martensite.	3.5	6.3	1.5	2.5
30% Ni austenite..	0.6	0.6	0.25	15.5
30% Ni martensite.	0.6	0.6	0.25	15.5

To some extent the present work also suffers from uncertain factors controlling the absorption rate. However, the averaging procedure of drawing the best line through the absorption concentration as a function of time is more reliable than the method employing single values obtained from permeation experiments. The best procedure, used by Hill and Johnson[25] for nickel and more recently for iron, involves a determination of evolution rate, independent of initial concentration and probably unaffected by conditions at the out-going surface. The value obtained at room temperature by Hill and Johnson,[26] for annealed iron, is 2.1×10^{-7} cm²/sec. Results here generally agree with this value.

It appears that increasing nickel concentration in ferrite decreases the diffusivity of hydrogen. Transformation of 10 percent nickel ferrite to martensite of the same composition increases the diffusion rate considerably. In view of the apparent alloy composition dependence of diffusivity in ferrite, the value for austenite at 30 percent nickel evidently indicates that the structural effect of austenite, relative to ferrite, is small. Finally, transformation of 30 percent nickel austenite to martensite appears to decrease the diffusivity, while the

saturation concentration is greatly increased.

However, though the preceding diffusion constants are of the correct order of magnitude, many uncertainties remain to be explained. The atom fraction of hydrogen in iron is small and, accordingly, the diffusion constant could be assumed to be independent of composition in hydrogen. But values calculated from absorption data in hydrogen sulfide, Table 6, generally are larger than values calculated from cathodic charging data, Table 5. Furthermore, the apparent effect of structure is not consistent with respect to both sets of data. Obviously, it will be necessary to reexamine this problem with a more refined technique in order to provide more precise data. In particular, the temperature dependence for the various structures and conditions must be obtained to separate the structural contribution from the activation energy. For the present, the values in Tables 5 and 6 appear to offer a reasonable first approximation.

Hydrogen Concentration and Fracture

Previous results have indicated that spontaneous fracture of a deformed specimen exposed to hydrogen sulfide is dependent on both hydrogen concentration and mechanical properties. This conclusion is substantiated further by considering the data for 10 and 30 percent nickel steel in the martensitic state. Both alloys have the same mechanical properties, R_c 27-28, but only the 10 percent nickel martensite fails when exposed to hydrogen sulfide. Present data show that the hydrogen contents of the two alloys are 3.5 and 0.6 ml/100gm respectively. Under conditions of cathodic charging both martensitic alloys fail and the results show that the hydrogen content of the 30 percent nickel martensite when cathodically charged at 2 ma/in.^{2} is increased by a factor of almost 50, relative to hydrogen sulfide. In this example direct comparison of failure susceptibility is possible between similar structures of the same mechanical properties but different hydrogen contents. The conclusion that hydrogen is primarily responsible for failure seems inescapable.

The one-atmosphere, isobaric measurements made at high temperatures show that the equilibrium hydrogen concentration in austenite is greater than in ferrite. Extrapolation of these data to room temperature indicates that the transformation of saturated, retained austenite should liberate hydrogen which may be the cause of failure. Present data do not sustain this hypothesis. Under cathodic charging relative hydrogen content is reversed from that found by extrapolation of high temperature data while in hydrogen sulfide, the amount of hydrogen absorbed is identical in both structures.

The effect of internal transformation stresses on the hydrogen content appears to be small. This is indicated by the identical concentrations observed for 10 percent nickel ferrite and martensite in hydrogen sulfide or cathodic charging conditions. The same identity is observed between annealed carbon steel and carbon martensite.

The only alloy in which a direct structural comparison could be made was 30 percent nickel austenite and martensite. If, based on the preceding evidence from carbon steel and 10 percent nickel steel, internal stress is not a significant factor, then the face centered cubic structure (austenite) absorbs less hydrogen than the body centered cubic structure (martensite) under cathodic charging conditions. This is contrary to expectation, based on heat of solution in pure iron and on one atmosphere isobaric measurements at elevated temperatures. But extension of these data for iron to a 30 percent nickel iron alloy and to conditions of extremely high effective pressure certainly is questionable.

Effect of Chemical Conditions on Hydrogen Absorption and Fracture.

The most striking effect of chemical composition of the aqueous phase is the fifteenfold increase in hydrogen absorbed from sulfuric acid as a result of a small addition of carbon disulfide. While the state of sulfur in 4 percent sulfuric acid is not certain, it is probable that sulfur in the acid and the sulfide ion in hydrogen sulfide have similar effects on hydrogen absorption. Thus, for a given level of mechanical properties (stress) failures in hydrogen sulfide may be reasonably associated with the large hydrogen concentration promoted by the sulfide ion.

The hydrogen concentration increases systematically with current density and follows the same relationship as hydrogen overvoltage. For the present purpose, this relationship provides a means of systematically changing the hydrogen concentration in fracture experiments and, since the saturation concentration is constant in time, the experiments may be conducted at constant hydrogen concentration throughout the period of the test.

Because failure under constant deformation frequently occurs before the specimen is saturated, it appears that it is necessary only to reach some level of hydrogen in the outer surface layers. It is impossible to set a limiting hydrogen concentration or stress level below which fracture will not occur when both hydrogen gradients and stress gradients exist. It is necessary to conduct experiments uniform in time, stress and hydrogen concentration across the section. In other words, it is necessary to investigate failure as a function of stress, under conditions of hydrogen saturation and as a function of hydrogen concentration at saturation by varying the current density.

Summary of Factors Controlling Hydrogen Concentration

1. For immersion in hydrogen sulfide, the principal factor determining the saturation concentration of hydrogen is the alloy composition. The observed decrease in hydrogen concentration with increasing nickel content apparently is due to the decreased corrosion rate (decreased effective hydrogen pressure); but, the fact that the change in hydrogen concentration is not proportional to the change in alloy composition, indicates that some other factor is involved. This conclusion is further substantiated by the difference in hydrogen content observed in Armco iron and in carbon steel where the corrosion rates are substantially the same.

2. The same concentration of hydrogen is absorbed from hydrogen sulfide by ferrite and martensite, or austenite and martensite of the same composition.

3. Under cathodic charging conditions, the transformation of 30 percent nickel austenite to martensite results in a three-fold increase in hydrogen concentration in the latter structure. No such change is observed for the 10 percent nickel ferrite and martensite structures.

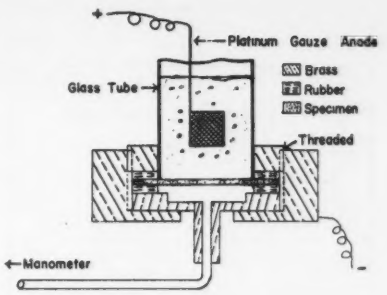


Figure 30—Hydrogen permeation apparatus.

4. The hydrogen concentration absorbed from cathodic charging in sulfuric acid, with or without a sulfide poison, is related to the current density in the same way as hydrogen overvoltage, namely:

$$[H] = a + b \log (C.D.)$$

5. A small amount of carbon disulfide in sulfuric acid increases the hydrogen content absorbed at a current density of 2 ma/in.^{2} by a factor of 15.

Correlation of Hydrogen Content And Spontaneous Fracture

1. The hydrogen content of 30 percent nickel martensite in hydrogen sulfide is low and failure does not occur under constant deformation. When cathodically charged to a higher hydrogen concentration, failure invariably results.

2. Failure does not occur in the nickel steels when charged in sodium hydroxide, apparently because of the low hydrogen concentration. The more susceptible carbon martensites fail instantly when charged but do not fail when immersed in sodium hydroxide without an applied current.

3. Considering the accumulated results of cracking experiments and the present data on absorption, it appears that failure depends on two conditions: a) the hydrogen content and, b) the stress level.

4. Present evidence indicates that the primary function of corrosion is to supply an effective cathodic current for the production of hydrogen, part of which is absorbed. Failure is a consequence of the hydrogen absorption.

Part 3—Hydrogen Permeability of Austenite, Martensite and Ferrite in Iron-Nickel Alloys

Introduction

Passage of hydrogen generated from an aqueous solution through a metal diaphragm involves at least four separate processes:

1. Reduction of hydrogen at the metal surface,
2. Its absorption by the metal,
3. Diffusion through the diaphragm and,
4. Evolution of hydrogen gas at the exit side.

The diffusion process controls the rate of passage of hydrogen after it has been absorbed at the surface. However, the transport of hydrogen is proportional to the concentration gradient existing across a diaphragm and, accordingly, it is dependent on the hydrogen concentration at the entrance and exit sides. The fraction of hydrogen produced at a

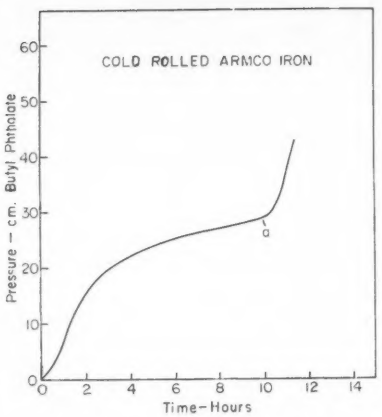


Figure 31—Effect of poison on hydrogen permeation in cathodic charging. At beginning of run, only residual amount of poison is in electrolyte. After initial rise, permeation rate soon falls due to poison depletion. At point *a* a few drops of poison were added to electrolyte.

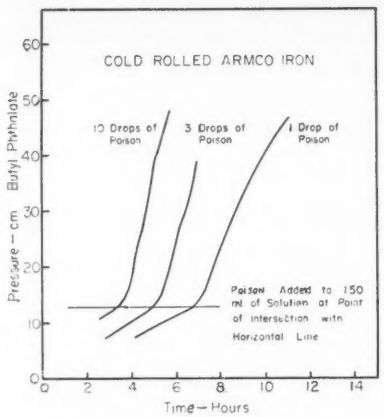

COLD ROLLED ARMCO IRON
10 Drops of Poison
3 Drops of Poison
1 Drop of Poison
Poison Added to 150 ml of Solution at Point of Intersection with Horizontal Line
Pressure – cm. Butyl Phthalate
Time – Hours

Figure 32—Quantitative effect of poison on permeation rate of hydrogen. Graph shows abstracted portions of one continuous run. Steady state flow was achieved in pure 4 percent sulfuric acid, one drop of poison added; effect of poison was allowed to decline between additions. No further effect was noticed beyond three drops.

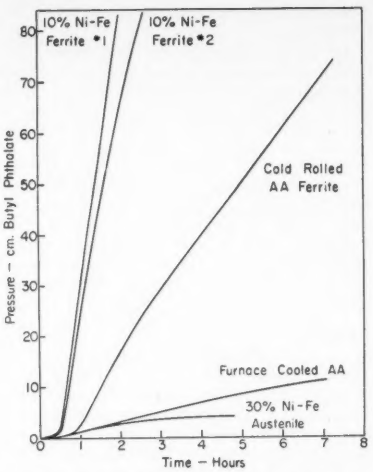


Figure 33—Permeation of hydrogen through ferrite and austenite, cathodically charged in sulfuric acid at 125 ma/ sq. in.

particular current density that actually enters the metal is small and is extremely dependent on surface conditions. Thus, experimental conditions are not well defined and the apparent diffusion constant obtained from permeability experiments in aqueous solutions is correspondingly inaccurate.

In the present work, it was important to establish whether solubility and permeability data could be correlated for the different structures and whether different methods of calculation were consistent with each other.

The permeability (ml of hydrogen/ cm^2/sec.) is defined by the expression

$$P = D \cdot \frac{C_i - C_o}{d}$$

where D is the diffusion constant (cm^2/sec); C_i and C_o are the hydrogen concentrations (ml/cm^3 of iron) at the in-going and out-going sides of the diaphragm, respectively; d is the thickness of the diaphragm (cm). Assuming a negligible solubility at the exit side where the hydrogen pressure approaches zero, the permeability reduces to:

$$P = D \frac{C_i}{d}$$

It may be assumed that C_i is identical with the saturation solubility previously determined for the same materials and conditions.

Diffusivity also may be determined from the time required to reach a constant rate of passage of gas through a diaphragm. In practice, the rate of evolution of gas, measured at the exit side, increases from zero to a constant rate and the diffusivity, D, is inversely proportional to the time, t, required to reach a steady state and to the square of the thickness of the diaphragm:[24]

$$D = \frac{d^2}{6t}$$

This method has the same uncertainties as the previous method involving the hydrogen concentration. The time to attain steady state, measured from the time at which cathodic current is started, is similarly dependent on surface conditions that are difficult to control or evaluate.

Experimental Procedure

Materials investigated were low carbon steel (0.04 percent C), 10 percent nickel-iron and 30 percent nickel-iron alloys. The two nickel alloys were from the same stock used for hydrogen absorption and fracture experiments.

Heat treatment was conducted in air at 900 C. As in previous work, slow cooling of the 10 percent nickel alloy produced a ferritic structure and quenching to room temperature resulted in a 100 percent martensitic structure. The 30 percent nickel alloy was completely austenitic at room temperature (Ms at —50 C) and 90-95 percent martensitic after transforming at the boiling point of liquid air (—190 C). The low carbon steel was used in the cold rolled condition and after annealing at 900 C.

After chemically or mechanically removing oxides resulting from heat treatment, all surfaces were electropolished. The electrolyte was 5 percent perchloric acid (70 percent $HClO_4$) in glacial acetic acid. A current of 0.4-0.5 amps/in^2 was used for five minutes at 15 C to produce a bright and clean surface.

Diaphragm type experiments were made in which one side of a specimen was cathodically charged with hydrogen and the hydrogen that diffused through the specimen was collected and measured in a manometer on the opposite side. The apparatus, shown in Figure 30, is essentially that used by Darken and Smith[18] and others, but modified for cathodic charging. The circular disk specimen is set in a brass holder and each side is fitted with rubber gaskets so that when the two parts of the holder are joined the pressure on the rubber not only separates the two sides from each other but also forces the top rubber gasket against the glass tube making a water-tight connection to the specimen. Specimens were in the form of flat disks 2¾ inches in diameter and 0.0625-inch or less in thickness, with an effective exposed area of 2.4 square inches. The electrolyte was contained in the glass tube; a platinum gauze cylinder served as the anode, while the specimen was made cathodic by electrical connection through the holder. The bottom side of the specimen opened to a manometer using butyl phthalate as a liquid and measurements were recorded of the height of the meniscus in the manometer tube of calibrated inner diameter.

The electropolished specimen was first fitted and tightened into the holder; it was then tested for air-tightness by transferring the manometer connection to a vacuum pump and checking for a pressure rise after evacuation with an attached mercury manometer. When the apparatus was found to be sufficiently tight it was disconnected from the vacuum pump. To insure cleanliness of the surface, the specimen was then etched with a small amount of 1:3 nitric acid for 30 to 60 seconds, rinsed with distilled water and dried with alcohol. The specimen and holder were allowed to come to temperature equilibrium with the surroundings, after which they were reconnected to the butyl phthalate manometer. The temperature inside the cell was measured, 4 percent sulfuric acid electrolyte was brought to this temperature and 150 ml of acid were poured into the glass cell. When the manometer level had stabilized, five drops of phosphorus dissolved in carbon disulfide were added to the electrolyte, the electrical circuit was completed to a 6-volt storage battery and the time of exposure was recorded from this moment. Current was maintained at 300 milliamperes, corresponding to a current density of 125 milliamperes per square inch. Readings of the height of the manometer were taken initially every tenth of an hour and less frequently as the course of the curve was established. No provision was made for controlling the temperature so that the test temperature was nominally room temperature which varied throughout the test from 21 to 27 C, although the range in any one test was not more than 2 or 3 degrees. The ambient temperature was recorded with each manometer reading.

Initial experiments to establish procedure and to study experimental variables were made with cold rolled mild steel, without electropolishing.

Effect of the phosphorus-CS_2 poison is shown in Figure 31. An electrolyte was used which had been decanted from a mixture of the 4 percent H_2SO_4 and the poison, so that there was no excess poison in the solution but only the small amount dissolved in the acid. The initial part of the curve of Figure 31 shows that the effect of the poison does not last long and the permeation rate soon declines. At point a a few drops of poison were added with an immediate effect. After about 30 minutes the rate was greater than that at the beginning of the test.

Further experiments to determine more quantitatively the effect of poison were made by adding known amounts of poison to an electrolyte of pure 4 percent sulfuric acid after steady-state flow of hydrogen had been achieved. An amount of poison was added, its effect was measured, and then the permeation rate was allowed to drop close to the original value before another increment of poison was added. Abstracted portions of curves showing only relative values for three different amounts of poison are shown in Figure 32. Poison was added at the point of intersection of the curves with the horizontal line. One drop of poison increased the permeation rate almost to that at the end of the curve in Figure 31. Three drops increased the rate slightly more while 10 drops did not produce any further increase. On this basis, the standard amount of poison used in later tests was five drops, which were replenished if the test ran for more than a few hours.

It was obvious from these results and those shown in Figure 31, that the effectiveness of the poison decreases in time. Thus, in any test using such a poison, it is important that its supply be maintained. On the other hand, it was also shown how little poison is required to produce the maximum possible effect.

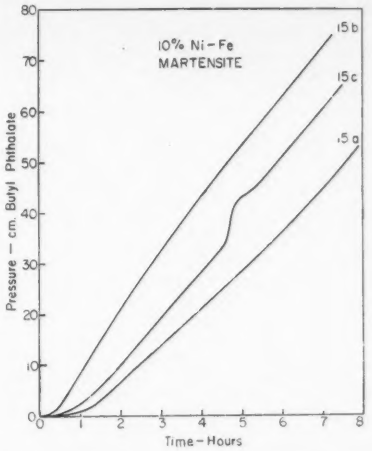


Figure 34—Hydrogen permeability of 10 percent nickel martensite and effect of surface treatment. All runs were made on the same specimen. Run *a* was surface electropolished, unetched; Run *b* was surface etched; Run *c* was made five days after Run *b* with no additional surface treatment.

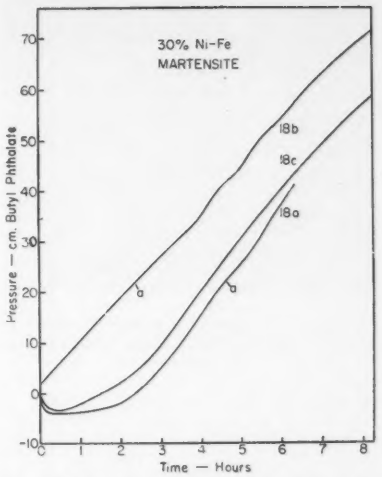


Figure 35—Hydrogen permeability of 30 percent Ni martensite. All data were obtained from the same specimen. Run 18b is a continuation of 18a, and 18c was obtained five days after 18b. Poison was added at points indicated by "a."

TABLE 7

Structure	C_s (125 ma/in²) (ml/gm)	Observed Permeation Rate (cm/hr)	Permeability x 10^6 (ml/cm²/sec)	$D = \frac{Pd}{C_s}$ (cm²/sec/ x 10^7)	$D = \frac{d^2}{6t}$ (cm²/sec/ x 10^7)
10 nickel martensite	0.13	13	28.5	24	30
10 nickel ferrite	0.13	50	110	90	17
30 nickel martensite	0.30	10	22	6	2.6
30 nickel austenite	0.13	(2)	(4.4)	(4)	—
Iron, annealed	0.50	2	4.4	1	20
Iron, cold worked	—	16	35	—	16

Results

Figure 33 shows the results of permeability tests on several of the materials, plotting manometer height versus the time of exposure. All runs were made under the conditions previously given, including electropolishing. The highest permeation rate occurs in the annealed 10 percent nickel ferrite structure. Reproducibility of results for different specimens of the same material is shown by the close agreement of two tests with this material. Cold rolled mild steel has the next highest permeability while the same material in the annealed state has a much lower permeability. This agrees with similar results found by Darken and Smith[18] after annealing cold rolled, 1020 steel. The permeation rate for the 30 percent nickel austenite appears to be the lowest of the group.

Permeability of the martensitic structure of the 10 percent nickel material is shown in Figure 34. It is somewhat lower than that for the ferrite structure of the same composition which is opposite to the relation found in the low carbon iron between the cold worked and annealed structures. This also brings out differences between the 10 percent nickel martensite and ferrite which were not indicated in the hydrogen absorption tests. The curves in Figure 34 relate to different runs on the same specimen. Another specimen showed approximately the same permeability. The first run, 15a, was made with the surface of the specimen electrolytically polished but not etched. Run 15b was started about 15 hours after the end of the first run but this time the surface was etched first with nitric acid. After this run the specimen was washed and dried with alcohol and then, five days later, run 15c was made without any further treatment of the surface. These variations appear not to have affected the steady-state permeation rate very much but the time lag before reaching steady state is significantly affected.

All remaining tests were made on the 30 percent nickel material and, because of lack of material, all on the same specimen. To check the previous results for 30 percent nickel austenite, the specimen was re-heat treated for ½ hour at 900 C and quenched in water. Oxide scale was removed, not without difficulty, with emery cloth. The specimen was electropolished again, removing a few thousandths of an inch in thickness, which made the thickness about 0.052-inch. The specimen, now in the austenitic state was rerun for hydrogen permeability under the usual conditions except that the surface was used as electropolished without etching in nitric acid. The results were practically the same as those found previously and shown in Figure 33, except that the initial rise occurred a little sooner. To be certain of the surface condition after the multiple heat treatments the specimen was further electropolished from a thickness of 0.052-inch to 0.042-inch.

This specimen was tested again for permeability in the austenitic state, after etching with nitric acid. Within one hour the height in the manometer had risen 7.0 cm., but it took the next four and one-half hours to rise to 9.3 cm., and from there on up to 73.0 hours, when the test was ended, it remained stationary.

After the last run the specimen was transformed to martensite by cooling in liquid air. Before transformation the hardness was Rockwell B50 and after transformation it rose to Rockwell B97, or C17.

This specimen, now as martensite, was tested for permeability under all the usual conditions with the results shown as curve 18a, in Figure 35. After 6.32 hours the manometer was disconnected from the permeability apparatus, the charging continued and the next morning the manometer was reconnected resulting in curve 18b in Figure 35. At points a in both curves an additional five drops of poison was added, apparently without significant effect. Curve 18b indicates that permeation was continuing at the same rate as first begun in 18a. This run was ended at 8.4 hours. The specimen was rinsed with water and alcohol and dried. After five days the specimen was again cathodically charged without any additional treatment, with the results shown in curve 18c of Figure 35. In spite of all precautions taken curves 18a and 18c exhibit an initial fall before the steady-state rate is established. The cause of this decrease in manometer height is unknown and unexplained.

Summary

Considering all the results, the 30 percent Ni austenite and the furnace cooled Armco ferrite were found to have the lowest permeation rates. This is consistent with results found by others for austenitic materials[27] and for annealed materials.[18] The 10 percent Ni and 30 percent Ni martensites and the cold rolled Armco ferrite appear to have similar, intermediate permeation rates. This agrees with the expectation that cold worked or structures containing internal stress would have higher permeabilities than annealed structures. The extremely high permeation rate for the furnace cooled 10 percent Ni ferrite, however, is entirely unexpected. This is equivalent to an annealed material and therefore, might be expected to have a low permeation rate. Obviously, the effect of the nickel addition must outweigh the effects of annealing. With the high permeability of the 10 percent Ni ferrite it is then rather difficult to explain the low permeation rate of the martensitic 10 percent Ni. Nosyreva[27] has reported a lower permeation rate for a 0.8 percent C martensite than for the same annealed material. In any case, these tests have shown that both structure and composition have a large effect upon permeation rate.

The diffusion constant, D, has been calculated from the time to attain steady state, or more specifically, the time extrapolated from the steady state rate to the time axis of the permeation rate curve. Because this time is affected by surface conditions, minimum time observed in several independent determinations has been used.

The diffusion constant has also been calculated directly from the relationship defining permeability and the saturation concentration of hydrogen, corrected for a current density of 125 ma/in^2. The observed steady state rate of permeation is converted to cm^3/cm^2/sec, the thickness of the diaphragm is taken as approximately 10^{-1} cm and the saturation concentration is converted to ml (hydrogen)/cm^3 (iron). All possible corrections for pressure change in the manometer, change in density with nickel concentration, etc., have not been introduced because they are not warranted in view of the errors inherent in the method. With the necessary conversion factors indicated above, the diffusion constants become:

$$D = 2.4 \times 10^{-5} \frac{P\,(cm/hr)}{C_s\,(cm^3/gm)}$$

The calculated quantities are collected in Table 7 along with the observed permeation rate.

Although many factors may reduce the permeation rate, it may be anticipated that none will increase it beyond the true value for a particular condition, so the highest permeation rate found was taken as representative of the true permeation rate for a particular metal.

Very little confidence should be placed in diffusion constants calculated by the time-intercept method. Figure 34, for 10 percent nickel martensite, demonstrates that different surface treatments produce diffusion coefficients varying by a factor of five. The lowest value of D is obtained by electropolishing and the largest value by etching immediately prior to charging with hydrogen. It may well be that the latter treatment presaturates the surface with hydrogen.

The diffusion constants calculated from permeation rate data are an order of magnitude larger than the constants calculated from absorption rate data. However, it must be remembered that the comparative current densities are also more than an order of magnitude larger in the permeation rate experiments and the correction applied to the saturation solubility may be inadequate. The only example for which all necessary data are available to calculate D from absorption experiments at nearly the same current density (100 ma/in^2) used in permeation experiments indicates that the two methods are in agreement, namely $D = 24 \times 10^{-7}$ cm^2/sec for 10 percent nickel martensite. Comparison of constants obtained from absorption data at 2 ma/in^2 and permeation data at 125 ma/in^2 indicates that the latter is ten times the former value of 2.4×10^{-7} cm^2/sec.

With respect to the comparative permeability of the different structures, the extremely high rate for the annealed 10 percent nickel steel is notable. Two independent experiments with this material produced essentially identical results. It also is significant that the two martensitic structures and the cold worked iron have approximately the same permeability. Data for the austentic structure indicate that permeability decreases in time to a small or negligible value; on the other hand, the permeability of the martensitic structure of the same composition is high and essentially similar to that of cold rolled iron or 10 percent nickel martensite.

Permeability is proportional to the product of the saturation solubility and the diffusion constant. Accordingly, even though annealed iron has the highest solubility, the permeation rate is among the lowest of all values measured because the apparent diffusion constant is small. It may be that the relative magnitudes of solubility and diffusion are responsible for the hydrogen blistering which is characteristic of annealed iron and mild steel. In iron-nickel alloys having a high permeability blisters appear only under extreme conditions. Similar observations have been made for cold rolled iron.

These experiments demonstrate again the enormous effect of surface active agents, carbon disulfide in the present example. The immediate rise in permeability after introduction of carbon disulfide presumably can be attributed to the observed fifteen fold increase in hydrogen concentration resulting from the addition of carbon disulfide.

Conclusions

As in the preceding investigation of solubility, the apparent diffusion constant calculated from permeability data varies widely with structure, chemical conditions of the metal interface and composition of the alloy. In general, values obtained from permeability experiments are an order of magnitude larger than the diffusion constants calculated from absorption data.

At present, the cause of the apparent variations in D is unknown. It is possible the assumption that D is independent of composition, implicit in all calculations, is incorrect even though the hydrogen concentration is small. It also may be true that D is not a single valued function of concentration but it is time dependent as a result of internal changes brought about by hydrogen absorption.

Apparent permeability is greatest for 10 percent nickel ferrite and least for annealed iron and 30 percent nickel austenite. Conversion of ferrite and austenite to martensite appears to produce the same intermediate permeation rate, approximately equal to that found for cold rolled iron.

Part 4—Delayed Fracture of Iron-Nickel Alloys Saturated With Hydrogen

Introduction

Experiments described in Parts 1-3 of this study have indicated that the failure of alloy steel in hydrogen sulfide is associated with the absorption of hydrogen resulting from the corrosion process. This final section is concerned with delayed fracture of 10 percent nickel steel in the ferritic and martensitic state, under static stress and saturated with different concentrations of hydrogen.

Previous results on fracture, hydrogen absorption and permeation tests have shown that the following conditions must be specified in order to obtain interpretable data from fracture experiments involving hydrogen:

1. To provide an independently variable stress, within the limits of creep and the tensile strength, fracture tests must be conducted under conditions of dead loading.
2. Because the time required to saturate with hydrogen may be a significant proportion of the fracture time, tests should be conducted on specimens pre-saturated with hydrogen and, therefore, without a hydrogen concentration gradient. Fracture time, measured from the beginning of immersion, cannot be interpreted for times of the order of the hours during which saturation is proceeding in specimens of the usual dimensions.
3. Since hydrogen is charged at a very high effective pressure and the saturation concentration is determined by this pressure, it is necessary to conduct fracture experiments under the conditions of charging; otherwise, when conducted in air, a concentration gradient exists across the specimen and, again, the state of the specimen is not well defined.
4. Because the saturation limit may be defined in terms of current density and chemical conditions, it is possible to evaluate the effect of hydrogen concentration on fracture. Effectively, fracture may be studied as a function of hydrogen concentration in a ternary alloy in which hydrogen is the third component.
5. The fracture stress of presaturated specimens has a fairly well defined meaning. Under these conditions plastic strain is small and the stress at fracture may be related to the true fracture stress.

In accordance with the preceding specifications, a test was devised to evaluate the effect of hydrogen on static fracture of 10 percent nickel ferrite and martensite of the same composition.

Materials and Experimental Procedure

Wire, 0.040-inch diameter, containing 10.30 percent Ni, 0.08 percent C, 0.07 percent Mn, 0.11 percent Si, 0.01 percent

S, 0.004 percent P, balance iron, was prepared for this work by the International Nickel Company.

Heat treatment at 1000 C was performed in a vertical tube furnace in argon purified by passage over titanium at 700 C. Wire specimens were prestraightened and supported in the furnace from a hanger carrying 12-16 specimens. To produce a ferritic structure, the wires were cooled slowly in the furnace over a period of 12-16 hours; a martensitic structure was produced by quenching into water at room temperature. Previous work with this alloy E, Part 1, showed that quenching produced 100 percent martensite, within the limits of the X-ray technique to detect retained austenite, about ±0.1 percent.

The heat treated wires were mounted in grips, attached to short lengths of chain to maintain axial loading and assembled in the hydrogen exposure apparatus, Figure 36. The wires passed through glass tubes provided with an inlet and outlet for hydrogen sulfide or exit for hydrogen in cathodic charging experiments. Both top and bottom of the tubes were closed with rubber stoppers; the wires passed through a greased hole in the upper stopper and through a tightly fitting hole in the lower stopper. Preliminary work with highly susceptible martensite in hydrogen sulfide showed that it was necessary to coat the extreme ends of the wires with a protective organic film to prevent failure due to bending stresses at the point of emergence of the wire from the lower stopper, as a result of the small curvature produced in quenching from 1000 C. Accordingly, all tests were conducted with wires nine inches between grips, of which a 6-inch length was inside the glass cells and a 3-inch length was exposed to the solutions. Stress was applied by loading a 4 to 1 beam with lead weights. The apparatus was calibrated by employing a spring balance in place of the specimen.

For cathodic charging, two platinum wire anodes were passed through the upper rubber stopper, parallel with the wire specimen. The electrolyte was 4 percent sulfuric acid, with or without a sulfide poison consisting of a few drops of carbon disulfide containing phosphorus.

The general procedure, determined by extensive preliminary experiments, involved presaturating the wires for a period of 24 hours at current densities of 2, 10 and 100 ma/in² while they were supported in the cell without applied load; after this period of saturation, the load was carefully applied by removing the support from under the weight hanger. Time was measured from this moment by electric counters connected to the levers in such a manner that when fracture occurred, they were automatically stopped.

Experimental Results

Experimental data obtained as described above, at three current densities, are shown in Figure 37 for the 10 percent nickel alloy in the ferritic condition.

Corresponding data for the same alloy in the martensitic condition, with and without a sulfide poison, are presented in Figure 38.

Fracture is obviously a function of stress, time and hydrogen concentration in an annealed (ferrite) structure. At the two lowest hydrogen concentrations it appears that a reasonably well defined "endurance stress" exists below which the material can support a stress indefinitely; however, some caution is indicated here because tests have been conducted for limited periods. At the higher hydrogen concentration, defined by a current density of 100 ma/in², the scatter in the data is considerable and an endurance stress is not well defined.

Nevertheless, it is instructive to correlate fracture stress with hydrogen content, which may be done in terms of the endurance stress defined above. For this purpose it will be assumed that the hydrogen concentration of ferrite is the same function of current density as that found for martensite, namely, $[H] = a + b \log I$. In view of the fact that the saturation limit for martensite and ferrite appear to be the same at 2 ma/in², the assumption seems to be warranted as a first approximation. Employing the derived hydrogen concentrations, it appears from Figure 39 that the endurance stress is a linear function of hydrogen concentration.

Recalling that fracture in the presaturated wires occurs without measurable reduction in area, Figure 22, the observed endurance stress may be related to the true fracture stress which, for the ferrite structure, is about 275,000 psi. Extrapolation of the straight line in Figure 39 to zero hydrogen indicates an apparent limiting stress of about 150,000 psi. However, it is reasonably certain that the indicated relationship is not linear to zero hydrogen concentration and the curve is probably concave upward, approaching the abscissa at zero hydrogen concentration at a much higher stress.

The relatively low values of the apparent endurance stress are very disturbing. Relative to the true fracture stress, it appears that the load carrying capacity of ferrite is reduced 75-80 percent by hydrogen absorption of the magnitude involved in these experiments and the load carrying capacity of martensite is destroyed almost completely.

The general form of the fracture stress-time curve for the 10 percent nickel alloy in the martensitic state, Figure 38, is the same as that for annealed ferrite, except that it is displaced to a lower level of stress. In this structure, the presence of internal transformation stresses makes it impossible to define a fracture stress in the sense used for the annealed structure, where applied stress and fracture stress are related directly. It appears that the applied stress and internal stress in martensite may be additive. With this assumption and taking the only instance where the hydrogen content of both martensite and ferrite are equal, 2 ma/in² with carbon disulfide, the apparent internal stress in martensite is equivalent to about 60,000 psi, relative to the annealed ferrite.

Data for both ferrite and martensite show another phenomenon that has not been observed previously, probably because it was hidden in the scatter of results in experiments of this general type. The dotted curves in Figure 37 correspond to very short failure times at the same stress as represented by the full-line curves for longer periods of time. The experimental points were generally obtained with sets of three specimens and, among the three specimens from the same wire and heat treatment, one specimen might fail almost instantly. It is important to notice in this connection, that the data are in two distinct groups and there are no points at intermediate times. This distribution of points strongly suggests that the effect is a property of the wire and not due to experimental variables. It appears that some specimens have defects of critical size and that failure of these specimens is almost independent of time. Microscopic examination to determine whether short-time fractures could be related to some visible, structural property were completely unsuccessful.

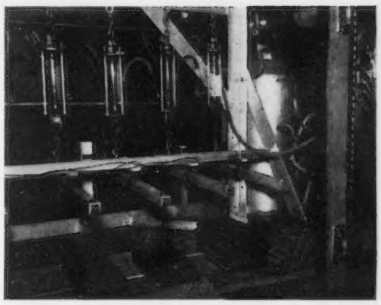


Figure 36—Apparatus for testing wire specimens under constant load in aqueous hydrogen sulfide, or saturated with hydrogen by continuous cathodic charging.

Summarizing Discussion of Delayed Fracture

Preceding results for materials saturated with hydrogen clearly show that failure is a well defined function of stress, time and hydrogen concentration. At stresses in excess of some lower limit, which is small in magnitude, the concept of "strength" appears to lose much of its significance because it is dependent on time. Also, the conventional definition of "embrittlement," measured by a ductility criterion obtained from a tensile test at a constant strain rate and varying stress, may require reinterpretation in terms of the integrated time at each stress. Certainly, numbers obtained from the test itself are not readily interpretable in terms of a mechanism.

With regard to structural considerations, it is clear that a fully annealed ferrite is susceptible to fracture, provided a sufficient stress is maintained or the hydrogen concentration is high enough. Results from constant deformation tests, in this instance, may lead to the wholly erroneous conclusion that this structure is not susceptible to failure.

The time dependence of fracture of martensite is similar to ferrite of the same composition. Apparent applied stresses at which fracture occurs are lower than for ferrite but this would be anticipated from the state of residual stress associated with the martensitic structure.

In view of present results it appears that the fracture process is a property of the iron-hydrogen system and not primarily dependent on a specific structure, or the transformation of retained austenite. Obviously the presence of martensite will result in failure at lower apparent stresses, but the structure itself is not a necessary condition of failure.

A fully austenitic structure was not investigated. However, apparent immunity of the austenitic steels may be questioned. First, hydrogen absorbed at room temperature is shown in Part 2 to be relatively little; second, the per-

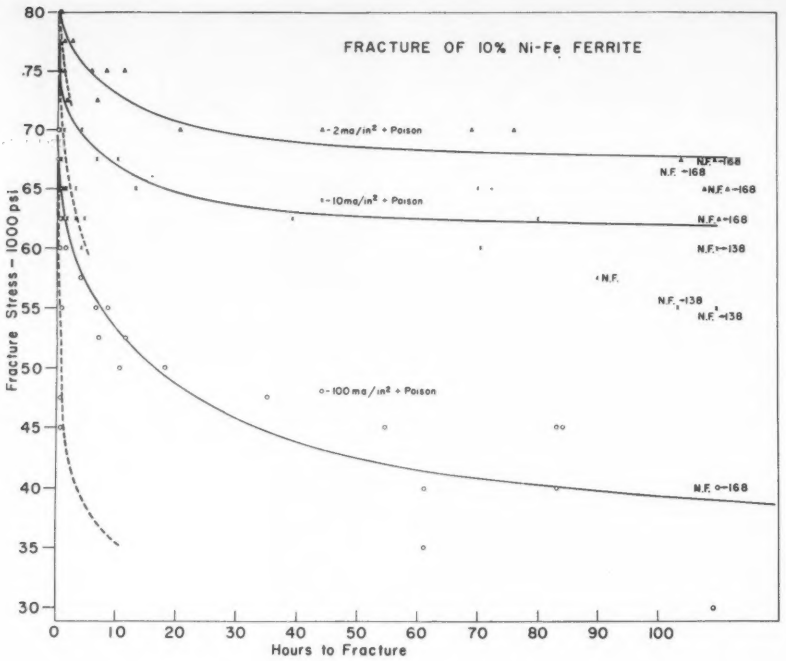


Figure 37—Fracture of 10 percent Ni-Fe ferrite at constant stress and constant hydrogen concentration, as a function of stress and time.

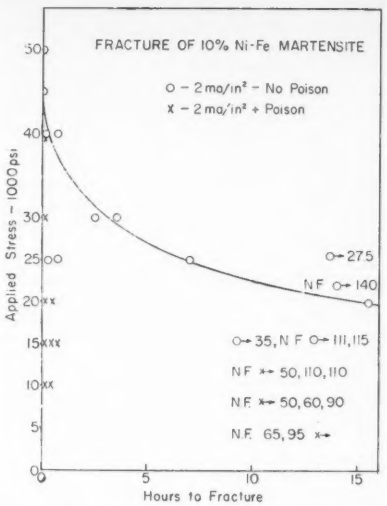

FRACTURE OF 10% Ni-Fe MARTENSITE
○ – 2 ma/in² – No Poison
x – 2 ma/in² + Poison
Applied Stress – 1000 psi
Hours to Fracture
○ - 275
N.F. ○ - 140
○ - 35, N.F. ○ - 111, 115
N.F. x - 50, 110, 110
N.F. x - 50, 60, 90
N.F. 65, 95 x -

Figure 38—Fracture of 10 percent Ni-Fe martensite at constant applied stress and two hydrogen concentrations, as a function of stress and time.

meation rate at room temperature also is small, or negligible, in accordance with a very small diffusion coefficient, of the order of 10^{-10} at room temperature.[19] Thus, the absence of failure at room temperature may be a consequence of the low hydrogen content and relatively low stresses applied in the usual constant deformation test. However, transgranular failure which is independent of heat treatment and composition, occurs in austenitic stainless steels in 42 percent magnesium chloride at its boiling point, 154 C and in other media at high temperatures. See, for example, Edeleanu.[35] It is suggested by the authors that the temperature increase may be sufficient to raise the surface hydrogen concentration, absorbed as a result of the corrosion reaction to the point where failure occurs by the same process as observed for ferrite and martensite. The same reasoning may apply to nickel which also fails at elevated temperatures and in which the diffusion coefficient is likewise small. Certainly, these possibilities should be investigated.

With respect to the significance of the corrosion reaction as a cause of failure through a stress corrosion cracking mechanism, present results appear to show that corrosion cracking is not a necessary part of the mechanism. For a given stress, failure time decreases with increasing cathodic current, contrary to the effect expected from a corrosion process, including the suppression of stress corrosion cracking by cathodic protection.[28] Thus, while it may be argued that corrosion is not completely suppressed in the range of current densities employed, the **direction** of change with current density is inconsistent with a corrosion process. Furthermore, in the present work, no structural evidence of corrosion cracking could be found.

Numerous proposals have been made regarding the detailed mechanism of cracking of iron saturated with hydrogen. Recent summaries[29, 30, 31] contain critical analyses of the various proposals. Similarity of the time dependence of failure with that found for glass is emphasized frequently. Frohmberg, Barnett and Troiano[30] compare their results on delayed fracture of quenched and tempered SAE 4340 steel with the data of Gurney and Pearson.[32] However, their curve of fracture stress versus log time, obtained for 4340 steel, is sigmoid in form whereas the corresponding curve for glass is a simple exponential function. The reason for the complex curve observed for 4340 steel may be ascribed to the fact that the fracture tests were conducted on unsaturated specimens, tested in air, and conditions were not constant throughout the test. In the present experiments the average results of fracture stress versus log fracture time are represented by straight lines over the whole span of time. See Figure 40.

Data on a variety of glasses obtained by Baker and Preston[33] over a time range of 10^7 seconds, indicate that a better representation is obtained by plotting the reciprocal of the fracture stress ("weakness") versus log time. Present results with steel are not sufficiently precise and do not cover a wide enough time interval, to discriminate between the two representations. When the data are plotted in terms of reciprocal fracture stress (actually, the dimensionless quantity E/F in accordance with the suggestion of Taylor[34]), the correspondence between wet, scratched Pyrex, dry Pyrex glass and the 10 percent nickel steels, Figure 41, is remarkable.

In particular, it is interesting that the strain at fracture, assuming uniform elastic strain, is about the same for glass and metal and equal to approximately 0.2 and 0.1 percent for ferrite and martensite, respectively. This strain may be associated with an amount of local plastic deformation required to nucleate a crack which is capable of rapid growth under the particular environmental conditions. Specimens that fail more or less instantly, at the same stress, may have preexisting cracks.

Delayed fracture of glass and metals presents two related problems: 1) the location and origin of fracture nuclei and 2) identification of the rate controlling process which defines the delay time. Because the mechanism of delayed fracture of glass is not yet clear and the apparent correspondence between failure in metals and glass has not been shown to derive from similar causes, it is premature and may be misleading to carry the analogy too far. In particular, it is clear from the effect of environment that fracture of glass originates at the surface; in metals containing hydrogen, fracture may also be initiated in the volume.

The present results show that for a given stress, fracture is a function of the hydrogen concentration. Accordingly, it may be anticipated that the location of fracture is associated with the distribution of hydrogen. In a steel containing residual hydrogen, fracture will originate in the volume. A partially saturated specimen, charged with hydrogen from the surface and subsequently tested in air, presumably will begin to fracture at some distance from the surface, depending on the time and conditions that define the location of the maximum hydrogen content. In fact, Toh and Baldwin[21] and Frohmberg, Barnett and Troiano[30] have shown that fracture can be propagated from the surface across a specimen in which hydrogen is concentrated only in the outer surface. In a saturated specimen without concentration gradients, as in the present experiments, fracture may be expected to originate at sites of stress concentration which can be present either at the surface or in the interior. The evidence from small wires with a martensitic structure shows that fracture originates at prior austenite grain boundaries and fracture cracks appear at both surface and volume locations. In an annealed ferritic structure, with a uniform hydrogen content and stress distribution, it is probable that cracks originate at sur-

face sites of local stress concentration and, accordingly, most failures in the present work exhibit a single fracture crack only.

In a martensitic structure, fracture clearly originates and propagates along prior austenite boundaries. The fracture nuclei in annealed ferrite have not been identified definitely. It is possible that nuclei are created during plastic deformation at the intersection of a screw dislocation and a slip plane, as suggested by Fisher.[39] Such nuclei normally would be unstable and tend to close rather than grow. However, if filled with hydrogen, the resulting pressure will prevent closure and the void may grow under an applied stress. Equilibrium pressures of hydrogen in steel are very high, no matter how they are calculated (see, for example, De Kazinczy[40] and Darken and Smith[18]) and the amount of hydrogen necessary to produce a high pressure in these sub-microscopic voids is correspondingly small.

Identification of the rate controlling process determining the time of fracture at constant stress also is not definitely established. Surface energy of a growing crack is lowered by adsorption of gas and, according to Orowan, the time delay for fracture of a brittle solid (glass and mica) is associated with the time for an adsorbing constituent to find its way to the leading edge of a growing crack where it is adsorbed. Gurney[37] suggests that the time for chemical reaction at the crack tip, rather than adsorption, is responsible for the time dependence. Taylor[34] proposes time-dependent rearrangement of bonds (in glass) at the site of fracture. In connection with the effect of hydrogen in metals, Petch and Stables[38] propose that hydrogen is adsorbed on the surface of a growing crack, lowering the energy necessary to propagate a crack; presumably, the time dependence of fracture originates in the time dependent diffusion of hydrogen from solution and the driving force for diffusion is ascribed to relief of strain energy. But, since there is no good evidence for dilation of the metal lattice by hydrogen, it appears that migration must be attributed to a different cause.

When the metal is saturated with hydrogen and saturation is maintained, as in the present experiments, hydrogen in the fracture nuclei is in equilibrium with hydrogen in solution and hydrogen at the outer surface. It has been shown that a definite concentration is required for fracture at a given stress (the endurance stress) and that the time for fracture at a stress in excess of the endurance limit is also dependent on the concentration. The minimum concentration could be associated with the pressure required to prevent closure of the fracture nuclei, at a given applied stress. Extension of the fracture nuclei lowers the pressure, requiring diffusion of hydrogen to reestablish equilibrium. With this model, the time required for fracture is associated either with time-dependent plastic-deformation (flow) under constant external stress or with diffusion of hydrogen, whichever is more rapid. The necessary strain data are not available to evaluate quantitatively the deformation hypothesis. Likewise, the temperature dependence of rate **at constant hydrogen concentration,** necessary to evaluate the diffusion hypothesis, is unknown and would be difficult to obtain. However, fracture time is an exponential function of the stress and creep rate at low temperatures also is an exponential function of stress[42,43] which appears to indicate that the rate limiting process may be the extension of fracture nuclei by deformation rather than hydrogen diffusion, under conditions of constant applied stress and hydrogen saturation.

Summary and Conclusions

There are several proposed mechanisms for sulfide cracking, all with some apparent support in theory and service experience. Since remedial measures depend on an accurate definition of mechanism and the significance of numerous variables, the present study was, to a large degree, concerned with an evaluation of different proposed mechanisms and a study of some of the more important variables.

As a result of this work, it now seems definite that the basic cause of failure in sulfide environments is associated with the absorption of hydrogen.

Although other factors may have an auxiliary effect, the principal factors determining failure in service are internal stress, the magnitude of applied stress and the hydrogen content.

Structure

Both martensitic and ferritic structures fail when the stress level and the hydrogen content are sufficiently high. Cold working also increases the susceptibility of ferrite to failure. Failure has not been produced in austenite but, before an absolute statement can be made, it will be necessary to conduct tests at constant stress, under dead loading conditions and at a temperature at which hydrogen can diffuse at a significant rate.

There is no evidence that retained austenite is a necessary part of the mechanism except that, if present, its transformation would provide some proportion of untempered martensite which would be more or less susceptible to failure, depending on carbon content. Recent evidence[41] suggests that a considerable proportion of martensite is required to affect significantly the fracture behavior.

Hydrogen absorption from hydrogen sulfide does not depend on structure, provided composition remains constant. Saturation limit is the same for a ferrite-pearlite aggregate and for the corresponding martensite in two carbon steels, 10 percent Ni ferrite or martensite and austenite or martensite in a 30 percent Ni steel. This equality of saturation concentration for different structures also is found when hydrogen is applied by cathodic charging, except in 30 percent Ni austenite and martensite. In the latter alloy, martensite absorbs about three times as much hydrogen as austenite.

Hydrogen permeation rate is dependent on structure. It is a minimum in austenite and annealed Armco ferrite; it is greater, and approximately equal, in 10 and 30 percent Ni martensite and in cold worked Armco iron; it is exceptionally high in 10 percent Ni ferrite.

Alloy Composition

Carbon, oxygen and nitrogen, as trace elements in 10 percent Ni steel, have no detectable effect on failure, down to a limit of about 0.002 percent.

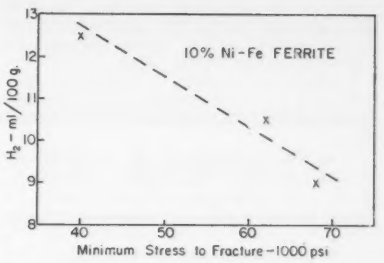


Figure 39—Relation between apparent "endurance stress" and hydrogen concentration in 10 percent Ni-Fe ferrite.

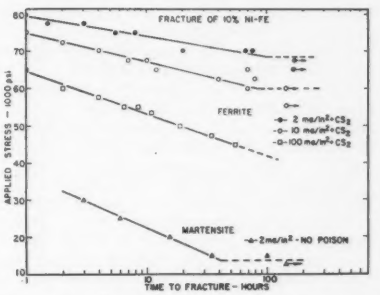


Figure 40—Delayed fracture of ferrite and martensite in an iron-nickel alloy under constant applied stress and constant hydrogen (saturation) concentration.

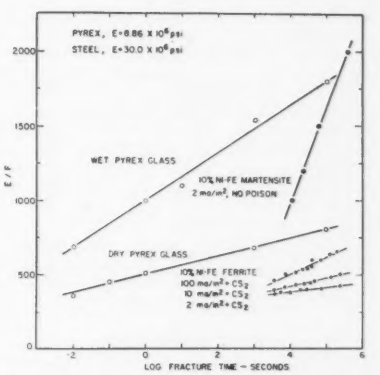


Figure 41—Comparison of delayed fracture in glass[33] with the corresponding time-dependent fracture in an iron-nickel alloy saturated with different concentrations of hydrogen.

The most pronounced effect of adding nickel to iron is a substantial increase in permeability to hydrogen. For both hydrogen sulfide exposure and cathodic charging the greatest hydrogen absorption is observed in Armco ferrite. Alloying with carbon or nickel decreases the saturation quantity with the exception of 30 percent nickel martensite which absorbs almost as much hydrogen as Armco ferrite when cathodically charged. In hydrogen sulfide absorption is largely dependent on corrosion rate which is determined by composition. However, the results in cathodic charging indicate that carbon and nickel have an absolute effect on hydrogen absorption.

Stress-Dependence of Failure

Tests conducted under conditions of constant stress and hydrogen concentration conclusively show that fracture of ferrite or martensite is dependent on stress, hydrogen concentration and time.

Above an apparent limiting "endurance stress," fracture is time dependent in the manner of the "static fatigue" of glass and other brittle solids.

Endurance stress is inversely proportional to the hydrogen concentration in the range of concentration investigated.

A model is proposed for the fracture mechanism under conditions of constant stress and hydrogen concentration. In terms of this model, the kinetics of the fracture process are associated with the stress and time-dependent extension of fracture nuclei.

Mechanical Properties

Significance of limiting strength or hardness, below which fracture does not occur in a constant deformation test, is evidently related to the endurance stress found in constant stress tests.

Cold working increases the saturation concentration of hydrogen and the internal stress, with the result that failure is promoted in a constant deformation test.

Interpretation of constant deformation tests is severely limited with respect to predictions about service life where loading conditions may result in local excessive stress and cause failure not anticipated from constant deformation tests.

Chemical Environment

It has been, shown that a trace of sulfide ion in aqueous solution results in a 15-fold increase in the saturation limit of hydrogen absorbed by a nickel steel. Because the quantity of hydrogen involved is more than sufficient to cause failure in time at a relatively low stress, the cause of catastrophic failures in hydrogen sulfide almost certainly may be associated with the promotion of hydrogen absorption characteristic of the sulfide ion. Similarly, increasing the hydrogen ion concentration of the environment increases hydrogen absorbed by the steel and promotes failure.

Tests conducted in the environment from which hydrogen is absorbed may differ appreciably from similar tests conducted out of the environment.

Recommendations

Evidence showing the dependence of fracture of steel on hydrogen, stress and time appears to indicate that there is no immediately obvious remedial measure that can be adopted with any assurance of immunity to failure under service conditions where hydrogen absorption may be anticipated and high operating stresses are involved.

Because it is impractical to lower stress significantly below the apparent "endurance limit," the remaining possibility involves the reduction of the hydrogen concentration in the steel.

With respect to experimental evaluation of the susceptibility of materials available, which are needed for immediate service, the present work has shown conclusively that dead-load tests must be made in the environment before safe operating procedures can be defined.

Acknowledgment:

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This summary report is based on a thesis submitted by A. E. Schuetz to the faculty of the School of Engineering, Yale University, in partial fullfillment of the requirements for the Degree of Doctor of Engineering.

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Publication 57-18

NACE TECHNICAL COMMITTEE REPORT

Service Experience of Glass-Reinforced Plastic Tanks

Status Report of NACE Technical Unit Committee T-1J On Oil Field Structural Plastics*

THIS REPORT is the fourth in a series of reports giving service experience of glass-reinforced plastic tanks.[1, 2, 3]

Glass-Polyester Tank

Previous reports showed the circumferential measurements of a glass mat reinforced polyester 250-barrel oil field storage tank, both empty and full, during three winters and summers. As a comparison of empty and full measurements during the second and third summers showed little difference, the operator discontinued measurements on this tank.

In August of 1956, the tank suffered a tension failure at one of the staves. The failure was so extensive that it was necessary to junk the tank inasmuch as it could not be repaired. This tank had been in service since October 1952 or a total of 46 months.

Glass-Epoxy Tanks

Figures 1, 2 and 3 show the circumferential measurement of a glass-epoxy 250-barrel oil field stock tank and a 500-

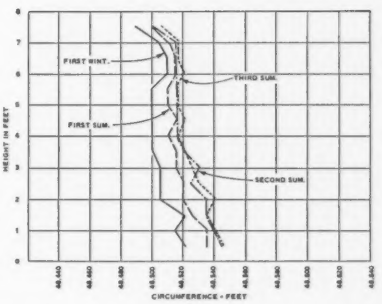

Figure 2—Circumferential measurements on an empty 250-barrel glass-epoxy tank.

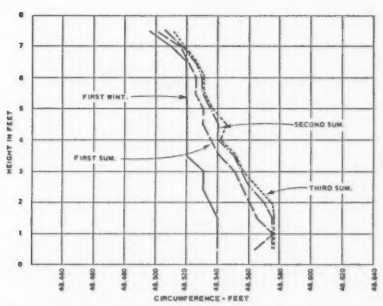

Figure 1—Circumferential measurements on a full 250-barrel glass-epoxy tank.

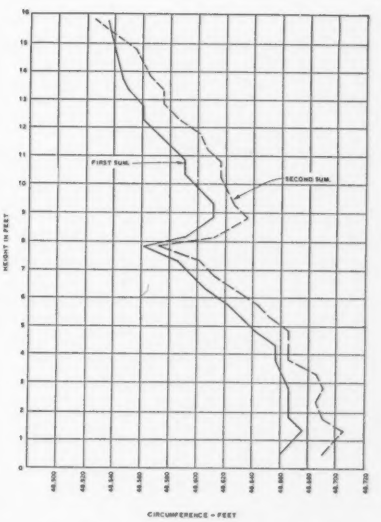

Figure 3—Circumferential measurements on a full 500-barrel glass-epoxy tank.

* Ward B. Sanford, Minnesota Mining and Manufacturing Co., Ridgefield, N. J., chairman.

Abstract

Previous reports had indicated that a glass mat reinforced polyester 250-barrel oil field storage tank showed little increase in circumferential measurement. It was found, however, that this tank suffered a tension failure at one of its staves and had to be scrapped after only 46 months' service.

Data are given to show the results of circumferential measurements made on a glass-epoxy 250-barrel oil field stock tank in both the full and the empty condition over one winter and three summers. Results also are reported of circumferential measurements made on a full 500-barrel wash tank during two summers. The average increase in circumference between the first and second summer for the 500-barrel tank was 0.020 as compared to 0.006 feet for the 250-barrel tank. **7.5.5**

barrel wash tank. A comparison of empty and full measurements during the second and third summers for the 250-barrel tank shows only a slight change. In view of these developments the operator believes that very little creep will continue. It may be noted here that the average circumferential increase from empty to full tank condition for the glass-epoxy tank is less than one-half that of the glass-polyester tank.

The strapping of the 500-barrel tank is presented here for the first time. The 500-barrel tank is similar to the 250-barrel tank with the following exceptions: (1) the tank is 16 feet high instead of eight feet high, and (2) the tank has an aluminum deck. The average increase in circumference between the first and second summer for the 500-barrel tank was 0.020 as compared to 0.006 feet for the 250-barrel tank.

The operator reports that he has eight glass-epoxy tanks in service and is well satisfied with this type of tank. The only deficiencies yet observed in any of them are (1) a few minor seam leaks such as are present on any bolted tank, and (2) occasional small seepages through some of the staves. These seepages do not yet appear to be of any real consequence.

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Publication 57-19

NACE TECHNICAL COMMITTEE REPORT

Experience With Oil Field Extruded Plastic Pipe in 1955

A Report of NACE Technical Unit Committee T-1J
On Oil Field Structural Plastics*

Abstract

The results of a questionnaire submitted to users of oil field extruded plastic pipe are summarized and evaluated. Fifty-six replies were received of which 16 were from independent producers and 40 from some six major producers. Data contained in the replies pertain to such matters as amount of extruded plastic pipe used by type of material, amount of pipe used by type of service, failure frequency, success in repairing leaks, distribution of failures according to amount of pipe used, cause of failure, reasons operators prefer extruded plastic pipe, and economic feasibility of increased use of plastic pipe. Replies received from independent operators and major companies are compiled separately in giving the above data. 7.2

Introduction

IN LATE 1955 NACE Technical Unit Committee T-1J undertook a study of current oil field experience with extruded plastic pipe. For non-users of plastic pipe the questionnaire was designed to find out what data they might need to aid them in utilizing this material. It was decided to gather these data through a questionnaire survey of oil industry members of NACE and non-NACE members who were known to be prominent users of extruded plastic pipe. The questionnaire was prepared, and after considerable review, was mailed in mid 1956 to about 250 individuals associated with oil production operations.

This report presents the results of this survey. Most questionnaire returns were received in August 1956; therefore, the results reported here should cover experience between the installation date in 1955 until August 1956. This ranged from 8 to 20 months.

Fifty-six replies were received. Sixteen were from independent producers while 40 were from six major producers. Seven major companies cooperated by forwarding replies to cover all of their United States operations.

Summary

1. The data gathered by this questionnaire revealed several operators used considerable footage of extruded plastic pipe in 1955 with no failures to date. These operators reported using extruded plastic pipe as a routine matter in low pressure service. In some cases this amounted to considerable footages. For instance, fifteen operators, who

* Ward B. Sanford, Minnesota Mining and Manufacturing Co., Ridgefield, N. J., chairman.

TABLE 1—Type of Extruded Plastic Pipe User

Type of User	Number of Independent Operators	Number of Major Companies	Total
Routine User...	10	14	24
Test User.....	1	16	17
Non-User.....	5	10	15
Total....	16	40	56

TABLE 2—Number of Extruded Plastic Pipe Installations by Type of Service

Type of Service	NUMBER OF INSTALLATIONS					
	Independent Operators		Major Companies		Total	
	Routine User	Test User	Routine User	Test User	Routine User	Test User
Low Pressure Flow Lines	143	..	94	23	237	23
Salt Water Systems	49	..	122	9	171	9
Gas	41	..	...	2	41	2
Other	..	1	8	6	8	7
Total	233	1	224	40	457	41
					Total 498	

TABLE 3—Amount of Extruded Plastic Pipe by Type of Service

Type of Service	FOOTAGE OF PIPE					
	Independent Operators		Major Companies		Total	
	Routine User	Test User	Routine User	Test User	Routine User	Test User
Low Pressure Flow Lines	236,515	....	171,141	32,901	407,656	32,901
Salt Water Systems	148,856	....	189,540	18,177	338,396	18,177
Gas	63,149	....	.....	4,900	63,149	4,900
Other	.....	1,000+	4,090	140+	4,090	1,140+
Total	448,520	1,000+	364,771	56,118+	813,291	57,118+
					Total 870,409+ feet	

Note: The plus sign represents pipe under evaluation by test users who did not report exact footage.

TABLE 4—Amount of Extruded Plastic Pipe by Type of Material

Pipe Material	FOOTAGE OF PIPE					
	Independent Operators		Major Companies		Total	
	Routine User	Test User	Routine User	Test User	Routine User	Test User
Butyrate	176,586	....	102,680	13,261	279,266	13,261
Styrene Copolymer	43,600	1,000+	186,801	22,760	230,401	23,760
PVC	170,334	....	71,240	20,097+	241,574	20,097
Not Identified	58,000	....	4,050	....	62,050	.....
Total	318,520	1,000+	364,771	43,151+	813,291	57,118+
					Total: 870,409+ feet	

Note: The plus sign represents pipe under evaluation by test users who did not report exact footage or type plastic.

TABLE 5—Failure Frequency According to Number of Routine Users, Number of Failures and Pipe Footage

	Independent Operators	Major Companies	Total
Number of Users Having No Failures	8	7	15
Pipe Footage Used with No Failures	428,435	239,570	668,005
Number of Users Having Failures	2	7	9
Number of Failures Experienced	5	17	22
Pipe Footage of Users Having Failures	20,085+	125,201+	145,286+

Note: Plus sign represents pipe under evaluation by test users who did not report exact footage.

TABLE 6—Failure Frequency According to Number of Test Users, Number of Failures and Pipe Footage

	Independent Operators	Major Companies
No. of Users Having No Failures	0	7
Pipe Footage Used with No Failures	0	30,370+
No. of Users Having Failures	1	8
No. of Failures Experienced	Several	34+ Several[1]
Pipe Footage of Users Having Failures	1,000	12,781+

[1] One operator reported several failures in each of two installations.
Plus sign represents pipe under evaluation by test users who did not report exact footage.

TABLE 7—Success in Operating Without Further Leaks According to Type of User

	Independent Operators		Major Companies	
	Routine Users	Test Users	Routine Users	Test Users
Number of Users Experiencing Failures	2	1	7	8
Number of Failures Experienced	5	Several	17	34+ Several[1]
Number of Users Making Successful Repairs	1	0	7	3
Number of Users Experiencing Additional Failures	1	1	0	6
Number of Failures Experienced by Users Having Repair Difficulty	1	Several	0	28+ Several[1]

[1] One operator reported difficulty in repairing several failures in each of two systems.

reported using extruded plastic pipe in low pressure service, used 668,005 feet in 1955 with no failures at the time of answering this questionnaire. The five largest users in 1955 laid 585,549 feet of pipe without a failure at the time of reporting.

2. Nine smaller operators, who use the pipe in low pressure service, reported twenty-two failures in 145,286+ feet of 1955 pipe installations. This is equivalent to one failure in slightly less than every 7,000 feet of pipe.
3. Users classified as testing extruded plastic pipe did not fare so well however. Although seven operators used 30,370+ feet without failure, eight other operators experienced over 34 failures in about 12,800+ feet of pipe. This is equivalent to one failure every 400 feet of pipe. Analyses of the reports on 30 of the 34 failures indicated the pipe could have been under conditions of low safety factor.
4. Thirty-six operators, who used 850,000 feet of extruded plastic pipe in 1955, reported installation cost data of plastic and competitive pipe. The average data indicate plastic is somewhat cheaper to install, although certain special conditions alter this situation. Twenty operators (using 682,000 feet of pipe) reported plastic less expensive to install; sixteen operators (using 174,000 feet of pipe) reported it to be more expensive than other types.
5. Data desired most by small or non-users of extruded plastic pipe are physical properties and installation costs.

Discussion

The breakdown of extruded plastic pipe users, test users, and non-users in 1955 is shown in Table 1. The number of installations by type of service is shown in Table 2. The amount of pipe by type of service is shown in Table 3. The pipe footage covered by the questionnaire according to type of material is shown in Table 4. Polyvinyl chloride pipe includes both types since many of the persons questioned were unsure of the type they had used.

An independent operator reported the largest footage use by one operator of 206,500 feet. Of this approximately 162,300 feet were PVC and 44,200 feet were butyrate pipe. An independent also was the second largest consumer. This operator reported using 130,000 feet of which 90,000 was butyrate and 40,000 was styrene copolymer.

A major company reported the third largest footage use in 1955 of all companies reporting. That company used 110,000 feet of which approximately 59,200 feet were styrene copolymer, 45,000 feet were butyrate and 4,800 feet were PVC.

The fourth largest user of those reporting was an independent operator who used 65,000 feet, most of which was not identified. Fifth largest extruded plastic pipe consumer was a major com-

pany which used 19,000 feet, principally styrene copolymer and about 6,000 feet of butyrate.

Failure Experience

Failure experience of those operators who used extruded pipe on a routine basis is shown in Table 5. Fifteen operators used almost 700,000 feet of pipe without a failure. Nine operators, using 145,000 feet of extruded plastic pipe, experienced 21 failures. Among the latter group this is approximately one failure for every 7,000 feet of pipe used. For the complete group of routine users the failure frequency was approximately one per 39,000 feet of pipe.

The failure experience of those users who were testing extruded plastic pipe in 1955 is shown in Table 6. The 1,000 foot line installed by the independent operator, which experienced several failures, cannot be regarded as representative. The line was in a severe underwater service clamped to a weighted cable in a way to permit several types of stresses of unknown magnitudes. Seven major companies tested more than 30,000 feet of pipe without a failure. However, eight other operators experienced 34+ failures. Thirty of these were encountered by three operators, only one of whom reported the amount of pipe he tested. An analysis of these reports revealed one operator (experiencing seven failures) was operating a pipe thought to be polyethylene at a stress of approximately 900 psi at 90 F. The apparent reason for failure was reported as poor installation methods.

Another operator, who experienced six failures, reported data indicating failures occurred under the following conditions: To styrene copolymer pipe operating at stresses of 170-400 psi with a temperature of 90-95 F; to PVC pipe operating at a stress of about 270 psi at 90 F; and to butyrate pipe operating at a stress of 400 psi and 90 F.

The third operator, who experienced 17 failures on 8,800 feet of pipe, reported the failures occurred to styrene copolymer pipe at conditions amounting to a stress of about 670 psi and 105 F. In the cases of the last two operators the pipe wall thicknesses were not reported; standard weight thicknesses were used in calculating the reported stresses. The stresses would have been much higher of course if the pipe was actually thinner wall such as SWP.

The operator reporting several failures in each of two systems (see note on Table 6) was operating styrene copolymer pipe at a stress of about 550 psi and 80 F.

From this combination of actual and conservatively estimated stresses it appears that a large number of these failures occurred under conditions of very low safety factor. Thus, any pipe imperfections, or additional stresses of installation could easily cause a stress higher than the long term strength of the resin resulting in a failure.

Table 7 shows the success obtained by the various operators in repairing leaks. Six of the eight major companies reporting failures also had difficulty in operating without further difficulty. Four of these six operators were the same group mentioned above who were operating under conditions of low safety factor. The cause for further failures by the other two operators experiencing additional difficulty was not apparent from the reported data.

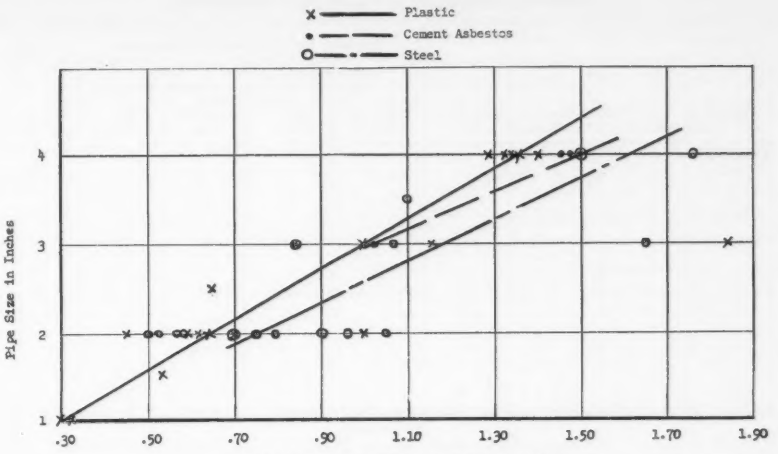

Figure 1—Installed costs of extruded plastic, steel and cement asbestos pipe.

TABLE 8—Cause of Failure as Evaluated by Operator

Cause of Failure	Independent Operators		Major Companies		Total
	Routine User	Test User	Routine User	Test User	
Excessive Pressure	0	0	0	6	6
Excessive Temperature	1	0	0	0	1
Poor Glue Joints	1	0	12	7	20
Poor Installation	1	0	1	4	6
Impact	1	0	0	2	3
Faulty Pipe	0	0	2	2	4
Other	1	Several	2	13+	16+
Total	5	Several	17	34+	56+

Note: Plus signs represent failures experienced by users who did not report the exact cause of failure.

TABLE 9—Distribution of Pipe Footage Used and Failures Experienced When Grouped by Fives From Largest to Smallest Pipe User

User Classification According to Amount of Pipe Used in 1955	Routine Users		Test Users	
	Footage	Failures	Footage	Failures
Top Five Largest Users of Pipe	585,549	0	....	....
Second Five Largest Users of Pipe	150,005	6	20,000	0
Third Five Largest Users of Pipe	35,101	10	21,967	4+
Fourth Five Largest Users of Pipe	27,940	3	....	....
Fifth Five Largest Users of Pipe	8,030	0	12,111	1
Sixth Five Largest Users of Pipe	5,510	3	2,920	+
Eleventh Smallest Users of Pipe	1,156	0	120+	29
Total	813,291	22	57,118+	34+
	Total Footage 870,409+			

TABLE 10—Identification of Failures by Type of Material, Location of Failure, and Part Failing

Identification of Failure	Number of Failures	
	Independent Operators	Major Companies
By Material:		
Butyrate	4	11
Styrene Copolymer	Several	25+
PVC	....	6
Unknown	1	7
Total	5+	49+
By Location:		
Above Ground	0	4
Buried	5+	45+
Total	5+	49+
By Part:		
Pipe Body	3+	23+
Coupling	1	22
Plastic-Metal Adapter	1	4
Total	5+	49+
Total Failures by Group	5+	51+

TABLE 11—Economics of Plastic Pipe Installations

Question: Can Plastic Pipe Be Installed Cheaper Than Pipe It Replaces	Answer: By Numbers of Operators				
	Independent Operators		Major Operators		
	Routine User	Test User	Routine User	Test User	Total
Answers:					
Yes	8		8	4	20
No	1		5	10	16
Equal	—		—	1	1
Depends on Situation	1		—	1	2
Total Operators Answering Question	10	0	13	16	39
Total Operators in Class	10	1	14	16	41

TABLE 12—Extruded Plastic Pipe Usage in Feet Versus Economics of Plastic Pipe by Operators

	Independent Operators		Major Companies		
	Routine Users	Test Users	Routine Users	Test Users	Total
No. of operators feeling plastic pipe cheaper than pipe it replaces	8	—	8	4	20
Plastic pipe footage used by above operators	379,290		274,990	28,450+	682,730+
No. of operators feeling plastic pipe more costly than other pipe	1	—	5	10	16
Plastic pipe footage used by latter operator	65,000		83,231	25,748+	173,979+

TABLE 13—Reported Reason for Using Extruded Plastic Pipe

Reason for Plastic Pipe	Number of Operators			
	Independent Operators		Major Companies	
	Routine Users	Test Users	Routine Users	Test Users
Corrosion Control	6	1	10	13
Paraffin Control	4	—	6	9
Flow Characteristics	2	—	2	1
Other			1	2

Note: Some operators reported duplicate reasons; others did not report a reason.

TABLE 14—Reasons for Not Using Extruded Plastic Pipe by Small and Non-Users

Reason	Number of Operators	
	Independent Operators	Major Companies
No field experience	2	7
No low pressure applications	2	14
Poor previous results	1	8
No contractor experienced	1	0
Insufficient physical data	3	12
Little cost savings possible	4	19
Other	2	2
Testing where appropriate	0	1

TABLE 15—Information Desired by Non-Users or Small Users of Extruded Plastic Pipe

Information Desired	Number of Operators	
	Independent Operators	Major Companies
Operation data on previous installations	6	19
Operating pressures for established and published safety factors	6	20
Installation costs	4	13
Other	2	3

Table 8 summarizes the cause attributed by the operator to the failure of his plastic pipe. The largest number of failures occurred among the major operators who were testing the pipe. Only seven failures were attributed to excessive pressure, whereas earlier in the report one operator reported seven failures with a material thought to be polyethylene operating at a stress of 900 psi at 90 F, and another reported 17 failures with styrene copolymer operating at a stress of 670 psi and 105 F. Yet only four of these 24 failures were thought by the operators to be due to excessive pressure. This suggests these operators might not have been familiar with stress limitations of extruded plastic pipe, and might have felt the failures must be due to something other than the strength properties.

Poor glue joints and poor installation can, of course, be corrected with installation experience. Impact failures fall in the category of operating technique which also is gained by experience. Only four failures were attributed to faulty pipe. Ten of the sixteen failures classified as due to other causes were said to be by split joints. Two failures classified due to other causes occurred to a butyrate installation operating above ground at 104 F.

An interesting aspect of the reported failures is the distribution of failures according to amount of pipe used. Table 9 is a summary of failures by users from largest to smallest after grouping the users by fives. These data roughly indicate 1955 failures are inversely proportional to the amount of pipe used during that year. It could also mean that the large users have had sufficient experience in previous years to enable them to use considerable footage in 1955 with few failures. Table 9, as well as Tables 5 and 6, indicate test users have a higher failure rate than those who use extruded plastic pipe as a routine matter in low pressure service.

Table 10 summarizes the failures by type of materials, location of pipe at the failure, and part of the pipe system experiencing the failure.

Costs

Table 11 shows a breakdown of how 41 operators felt about the installed cost of extruded plastic pipe versus other types of pipe. Thirty-eight of the 41 routine or test users expressed an opinion on their questionnaire. Table 12 illustrates the amount of pipe used by the 36 operators who definitely felt one way or the other on the question.

Nineteen of the 20 operators, who felt plastic pipe was less expensive to install than other pipe, reported cost figures. Figure 1 is a plot of pipe size versus the reported costs to install extruded plastic, steel, or cement asbestos pipe. This is a composite plot of all data reported with weighted average lines drawn in to indicate the average cost of the various types of pipe. The data reported indicate the average cost to install extruded plastic is slightly less than cement asbestos in the 3 and 4 inch sizes, and is less than steel pipe in the 2 to 4 inch size range.

Intended Purpose of Plastic Pipe

The questionnaire sought to determine the reason various operators used extruded plastic pipe. A summary of the replies is shown in Table 13. Many

operators reported duplicate reasons; others did not report any. Although the questionnaire did not inquire as to the success of plastic in controlling corrosion or paraffin, four operators voluntarily indicated plastic was not satisfactory for paraffin control.

Reasons For Not Using Plastic Pipe

The questionnaire also sought to determine why non-users or small users did not use extruded plastic pipe. Table 14 is a composite of these replies. Here again many operators reported duplicate reasons; others reported none.

Information Wanted by Small Users

This portion of the questionnaire was designed to determine what information small users or non-users would like in their consideration of extruded plastic pipe. A composite summary of the replies is shown in Table 15. The preponderance of operators desire data on strength characteristics backed up by operating experience, plus information on installation costs. The latter may be answered to some degree by the information presented in this report.

The five operators desiring other information were interested in (1) the technique of making road crossings for lines laid above ground, (2) paraffin control experience, (3) the best method for threading and coupling, (4) data on thermal expansion, and (5) impact strength.

Cathodic Protection of Internals of Ships*

By L. P. SUDRABIN

Introduction

THE COSTS of internal corrosion in ships have been established to be so great that the use of properly adapted corrosion control measures cannot be neglected. The magnitude of the problem can be readily seen from the examples cited below:

1. An API survey[1] has reported the costs of internal corrosion of an American Flag T-2 Tanker in clean products service to be conservatively $150,000 per year.
2. In super tankers[2] carrying Middle East crude, localized pitting on the bottom plates progresses at the rate of .125 inch per year. Steel heating coils are penetrated by pits in one year.
3. The repair and cleaning costs of one set of No. 3 deep tanks in a Liberty vessel are $7,650.00 per year.
4. The cost of manually removing rust scale from one set of deep tanks in a collier, in preparation for inspection, is $13,000.

There are four basic methods of corrosion control with which the ship designer or operator can reduce such losses:

1. Corrosion resistant materials
2. Protective coatings
3. Treatment of the environment
4. Cathodic protection

Each of the methods have special adaptabilities and advantages—none are cure-alls. In many cases the greatest protection may be obtained by a combination of the methods.

Although the cathodic protection principle has been used in the marine industry for more than 130 years[3] its greatest development has occurred within the past 10 years. This progress in the use of cathodic protection is attributable to the realization of the immense economic cost of corrosion, and the development of materials and methods of applying protective current. In some instances, cathodic protection is the one method by which the corrosion rate can virtually be reduced to zero.

Cathodic protection has been applied to internal tanks used for sea water ballast in several hundred vessels. Experience in more than 200 vessels (tankers in crude and clean products service, ore carriers, combination ore-petroleum products carriers, colliers, dry cargo vessels, etc.) has shown that the required corrosion control can be achieved by the coordination of the cathodic protection design and the ballast practices.

The advantages of cathodic protection for the corrosion control of internal tanks used for sea water ballast include:

1. Internal surfaces require no special preparation for cathodic protection to be effective.
2. Installation of anodes can be made during annual drydocking or in some cases by the crew while the vessel is underway.
3. Adequate cathodic protection will loosen (descale) previously formed rust and retard its further formation.

Corrosion Mechanism

The corrosion found in sea water ballasted tanks has two general forms:

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Abstract

Information obtained in connection with the application in ships of cathodic protection to internal tanks used for sea water ballast is discussed in detail. Particular attention is given to: (1) diagnosis of the corrosion problem, (2) design and application of protective current, and (3) measurement of the protection achieved. It was found that the required corrosion control could be achieved by coordination of the cathodic protection design and the ballast practices.

Advantages and disadvantages of panel tests are listed. Techniques used in evaluating the protection obtained on the internal bulkhead, skin and structural member surfaces by non-destructive testing are described. A corrosion mechanism is discussed briefly.

Cost of cathodic protection for adequate corrosion control of the internal cargo ballast compartments of vessels was found to range from $.02 to $.10 per square foot per year. 8.9.5

Figure 1—View of 32-pound shielded magnesium anodes secured to framing members of a dry cargo carrier.

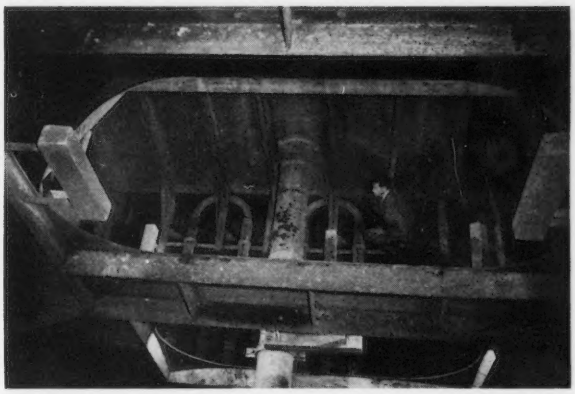

Figure 2—View of 90-pound magnesium anodes secured to framing members of combination ore-petroleum carrier.

TABLE 1—Anode Currents and Potentials

ANODE	Normal Ampere Hours per Pound	Normal Potential to Saturated KCl Calomel
Magnesium Grade A-H1	498	—1.50 volt
Galvomag	498	—1.65 volt
Zinc (<.0015% Fe)	335	—1.05 volt
Aluminum (3% Zn)	676	—1.00 volt

1. A comparatively uniform attack over all the tank surfaces is found in clean products cargo tankers, deep tanks of dry cargo vessels, tanks used solely for ballast in colliers, ore carriers, etc. Some examples of the severity of this corrosion include the replacement of bulkheads in six to eight years on tankers and within four years on ore carriers. Severe localized attack occurs at hard points where repeated flexure exposes an active iron surface.

   The corrosion product is an adherent rust scale (magnetic iron oxide).

2. Localized pitting (up to .125 inch/year) is found on the bottom and other horizontal surfaces including the upper surfaces of heating pipe coils in crude oil tankers. The attack is most severe in tanks ballasted each voyage.

Non-uniformities on the surface of iron produce galvanic cells comparable to dissimilar metal systems. Recent studies[4] show that mill scale (magnetic iron oxide) removed from the underlying iron is more cathodic than copper and requires more current for polarization to the protective potential of iron.

Rust scale removed from cargo ballast tanks is found to be as much as 0.2 volt positive to a saturated potassium chloride calomel reference electrode or nearly 1.0 volt cathodic to the potential of an active iron anode surface.

The intense localized pitting experienced on the horizontal surfaces of sea water ballasted crude cargo tanks is initiated by the settling of water on the horizontal surfaces displacing the protective oil film. Once the pit has formed, water tends to accumulate in it and the attack continues to progress. The corrosion cell established between the active pitting area and the adjoining partially oil impregnated rust (magnetic iron oxide)results in the migration of anions[5] into the pit causing a low pH (2.0-3.0).

The low pH of the corrosion products in the pit is not attributable to the character of the crude alone. The pH of a 6-inch layer of water under a stabilized Middle East sour crude cargo has been found to be 7.5 upon arrival in the United States. The pH of the products within the pits after emptying the cargo was as low as 2.0.

The author has observed[6] that as the rate of pit penetration increases, the pH of the core contents of the rust tubercles tends to decrease. For example, in a pit with a penetration rate of .10 inch per year, the core of the corrosion products will have a pH of less than 4.0. This low pH will be relatively independent of the pH of the bulk of the water and has been observed in the absence of the sulfates, sulfides, etc.[5] to which the low pH is often attributed.

Design of Cathodic Protection

The requirements of an effective cathodic protection design in a sea water ballasted tank are affected by all the variables of environment and operation which influence the corrosion mechanism.

Magnesium anodes are most frequently used as the protective current source. Each galvanic anode coupled to the tank is an independent protective system when immersed in sea water and does not require an external power source, insulated wire and fittings to function.

The low output voltage and secure anode attachment within the tank eliminate possible electrical failure and spark hazard. The galvanic anode product is not objectionable in the ballast water, whereas chlorine gas evolved on the surface of the non-sacrificial impressed current anode within the tank may introduce additional corrosion problems.

Magnesium and other galvanic anode materials may be designed to meet special service life and attachment requirements (see Table 1). Figure 1 shows 32-pound magnesium anodes in the deep tank of a dry cargo carrier. The anodes in this system are

Figure 3—Rust scale loosened by cathodic protection removed from a ballast tank after three months of operation.

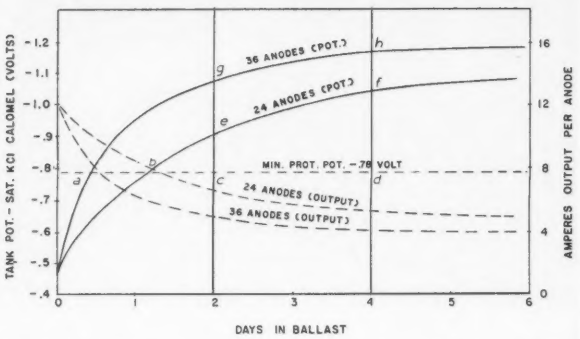

Figure 4—Relationship between anode number and tank potential to anode output.

secured to the structural members to resist damage by cargo handling. An insulating shield is used to retard the anode wastage caused by close coupling to the member.

In Figure 2 are shown 90-pound (77 pounds net) magnesium anodes with 15-inch brackets mounted in the wing tank of a combination ore-petroleum products vessel carrying Middle East crude. Such anodes are applied to tankers and in the tanks of other vessels which are used solely for sea water ballast. Good distribution of protective effect throughout a tank may often be made by attachment of the anodes to existing structural members.

The additional cost for special framing within a ballast tank to distribute the effect of the anodes can seldom be justified. In order to obtain the minimum protective effect in the recesses and corners of ballast tanks some excessive protective effect must occur on protuberant structural members.

Design Considerations

The cathodic protection system design must be adapted to meet the special requirements of the tank to be protected. The design considerations include:

1. Adequate protective current capacity to polarize the tank surfaces considerably beyond the minimum protective potential during the ballast period in order to loosen previously formed rust scale and to deposit a calcareous coating. Harvey and Streever[7] have indicated that a potential more electro-negative than —1.03 volt to a saturated KCl calomel reference electrode should be maintained and 4-5 ampere hours/sq ft of current applied to obtain 80-90 percent descaling of iron in sea water. The author's experience in ballast tanks shows that less ampere hours of current may be required for descaling and that such effects tend to be cumulative during repeated ballast, cargo and atmospheric exposure periods. Figure 3 shows the rust scale removed from the ballast tanks of a vessel within three months after cathodic protection was applied.

2. In short ballast period operation, more protective current (more anodes) must be used to quickly polarize the tank surfaces. Absorption of O_2 on the tank surface during atmospheric exposure causes a high current requirement immediately after submergence. For this reason, "booster" anode schemes to remove rust scale in the first months of protective system operation do not always yield adequate protective effects during the life of the protective installation.

3. Low salinity ballast water will increase the number and/or linear dimensions of the magnesium anodes needed.

Anode Output

The relationship between the number of anodes used, the output of each anode, the tank potential and the ballast time is illustrated in Figure 4.

The output of a 73-pound (60 pounds net) magnesium anode, immediately after ballasting, has been found to be about 12 amperes using a zero resistance ammeter circuit.

Since there is no external control, the output of the anode is determined by the driving voltage between the anode and the polarized tank. The driving voltage difference varies with time and the polarization characteristics of the ballast tank. For this reason, the design basis is most conveniently expressed as the square feet of tank surface protected by one anode of specified shape and material.

The initial output of twenty-four 73-pound magnesium anodes in a tank, just ballasted with sea water, is $(24 \times 12) = 288$ amperes; for thirty-six anodes it is $(36 \times 12) = 432$ amperes.

A comparison of the data in Figure 4 for two and four day ballast periods shows an example of the relation between the ballast period and the optimum number of anodes to be used in a protective system design.

The tank with 36 anodes is polarized to "g" in two days with an accumulation of ampere hour effect "a, c, g" above the minimum protective requirement. The protective potential is reached only in the second ballast day with 24 anodes and inadequate ampere hour effect "b, c, e" occurs to descale and coat the tank surfaces. Ballast water entrapped in the rust scale adhering to the tank surface will support a severely active corrosion system during the atmospheric and clean products cargo exposure periods.

The average individual anode output in a two day ballast period of the 24 anode installation is about nine amperes and for 36 anodes is about seven am-

peres. The 36 anodes needed for adequate protection would last about 28 percent longer than the 24 anode system.

The 36 anode accumulative effect "a, d, h" occurring in a four day ballast period will produce heavy calcareous salt deposits and represents excessive protective effect. The accumulative effect obtained from 24 anodes "b, d, f" is the more adequate design. The average individual anode output in a four day ballast period from a 24 anode system is about 7.5 amperes and from the 36 anode system about 6.0 amperes.

If a tank is ballasted with sea water 40 percent of the time, an anode system with four day ballast periods would last about 25 percent longer than with two day ballast periods. These observations were demonstrated in the wing tanks of two ore carriers operating in the Caribbean. The tanks of both vessels are ballasted 35 percent of the time.

Vessel No. 1 had two day ballast periods. One anode per 300 sq ft of tank surface did not effectively descale or coat the tank. One anode per 225 sq ft of tank surface was required for adequate protection.

Vessel No. 2 has five to six day ballast periods. One anode per 300 sq ft effectively descaled, coated and protected the tanks.

Aluminum Galvanic Anodes

Full scale tank studies are underway with aluminum galvanic anodes in ballast tanks of ore carriers. Early observations show that at least four times more aluminum anodes are required than magnesium to approach adequate protection with two day ballast periods. The initial output of each aluminum anode is about half that of a magnesium anode with the same dimensions. The output of the aluminum anode quickly decreases as the tank polarizes.

The limited driving voltage (—1.00 volt versus saturated KCl calomel) of aluminum requires the positioning of the anodes among framing members to obtain protection on all shielded surfaces.

The author's development in the use and the design of aluminum anodes for ballast tank protection is continuing because of the lower cost and weight per ampere year of protective effect.

Operation of Protective System

The performance of the cathodic protection system is dependent both on good installation practice and supervision of the ship's operators.

The best protection results are obtained by using a regular ballast plan (i.e., the same tanks are used for ballast on each voyage).

A rotated ballast plan may be used to include more tanks in the cathodic protection system by ballasting each tank every second or third voyage. The protection obtainable decreases with less frequent ballasting and longer atmospheric or cargo exposure periods.

The tanks should be pressed full to allow the protective current to reach all surfaces including the underside of the deck. Sloping vent lines should be installed at the high point in the cant of the deck to permit the escape of air to hatch trunk or vent. Hydrogen gas accumulation is minimized by pressing the tank full of ballast or keeping it empty.[2]

The tanks should be ballasted with pure sea water or with the brackish water for which the cathodic protection system was designed.

Anodes should be renewed as they are consumed.

Instructions for the operation of the protective system should be specially prepared for each vessel. Ballast report forms completed by the ships operators should be used for correlation of the performance of the cathodic protection system with the operation practices.

Anodes

Development of new anode materials and design have furnished the Corrosion Engineer with tools to expand the use of cathodic protection. Among the anode materials which furnish a protective current flow when coupled to iron are magnesium, zinc and aluminum. Properties of these anodes are tabulated.

The American Zinc Institute[8] through its research, has found that good zinc anode output can be obtained by limiting the iron content to less than .0015 percent.

Aluminum containing 3 percent zinc is available in cast forms for attachment to hulls and ballast tanks. This anode is a more recent development and full scale installations are being observed for performance.

The ship owner must be assured that the method used to control internal and/or external corrosion is effective by some meaningful measure.

Dr. R. M. Burns[9] has described the work of the Corrosion Engineer as the "—task of preserving the metallic state in surroundings where variables are complex, compounded and uncontrolled."

This complexity of corrosion is illustrated by seemingly identical ships in the same service and cargo compartments within the same ship which do not experience identical corrosion problems or even uniform distribution of attack throughout the same tank. The performance and the requirements of any of the basic methods of corrosion control vary from place to place and time to time. For these reasons, it is the author's opinion that the evaluation of the protective measures must be made of the actual structures individually by comparison of significant measurements at regular intervals.

Panel Tests

Prepared panels whose dimensions, weight, surface preparation and material can be accurately established, are used in laboratory and field corrosion testing. Since environment variables can be carefully controlled, panel studies have been very useful in screening corrosion control methods in the laboratory.

Advantages of panel tests include:

1. Systematic screening of methods (inhibitors, materials, applied current densities, etc.).
2. Quantitative measure of weight and dimension loss with time are easily made.

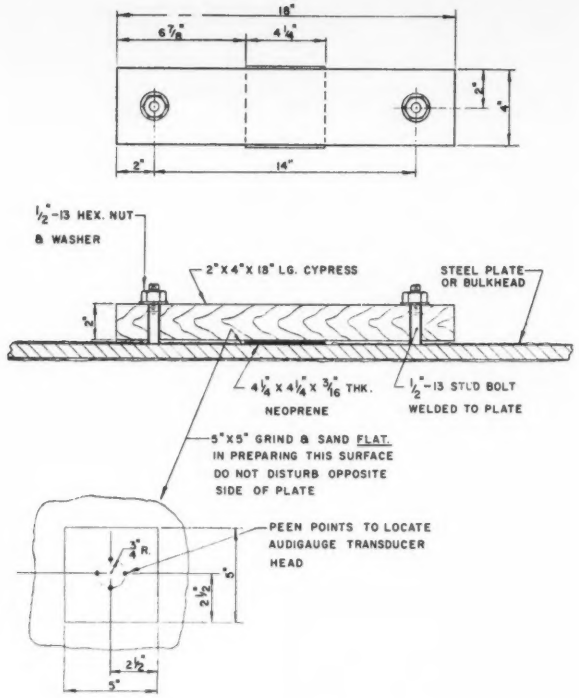

Figure 5—Audigauge station.

TABLE 2—T-2 Type Tanker Corrosion Rate Comparison With Audigauge Test Station Measurements

Tank Operation	No. of Tanks	No. of Test Stations	Average Penetration/Yr
C—CW	8	17	.0085″ (.0058″—.0135″)
C—CW—B	5	29	.017″ (.0115″—.0212″)
C—CW—B—CP	4	23	.0023″ (.0000″—.0058″)

Note: Ballast Time was 40%; Ballast Periods were 3—6 days.
Code to Letters is as Follows:
C —Clean Products Cargo
CW—Cold Sea Water Wash
B —Sea Water Ballast
CP —Cathodic Protection

3. Variables in the environment and control methods can be regulated.

There are certain limitations in interpreting panel tests made in the laboratory or within a tank and relating these observations to the corrosion control actually achieved. These limitations are as follows:

1. The surface condition of the panel and cargo compartment are not alike. For example, sand-blasted panels are usually anodic to the compartment.
2. Effects of working stresses are not produced on panels.
3. Sweating and condensation conditions are not easily reproduced on a panel.
4. Incidental areas of pitting on floor of sour crude cargo compartments are difficult to reproduce on a panel.
5. Short time studies on panels cannot always be extrapolated into anticipated long term results. For example Jupp[10] reports that the rate of metal loss increases as the members decrease in size because of working stresses.

Non-Destructive Testing

There are techniques presently in use for ultrasonic metal thickness testing. One type of equipment employs a variable frequency oscillator that is tuned until a standing wave of sound energy is set up. The frequency and wave length are related to the sound velocity and the thickness of material can be determined when the frequency is such that resonance is established. Scale or oil film on the opposite side of the steel plate is not included in the thickness measurement.

One procedure to evaluate the corrosion rate[11] in vessels is by annual surveys consisting of some 500 thickness measurements. Test locations are prepared by cleaning the metal surface and sanding it flat to accommodate the transducer head of the audigauge. This surface preparation and failure to repeat tests at precisely the same location on subsequent surveys limit the significance of the measurements. The apparent plate thickness may easily vary 10 percent on measurements made on a bulkhead 2 inches apart.

A practice, developed by the author, for measuring the effectiveness of cathodic protection on one surface is to prepare a permanent baseline surface on the opposite side of the bulkhead, on the deck, hull or member. The permanent baseline and audigauge transducer head location is protected by a pad between surveys (Figure 5). Although fewer thickness measurements may be made, they include all the factors influencing the protected system opposite the prepared audigauge testing surface.

A change of 1 percent in the plate thickness can be detected by a competent operator making measurements from the same surface. The technique in repeating measurements must be uniform including constant transducer head pressure, the same contact oil, observation of the frequency at the same harmonic, etc.

The fundamental frequency at which thickness resonance will occur for a material is expressed:

$$(1)\ f_1 = k/t$$

Solve for thickness "t"

$$(2)\ t = k/f_1$$

Where f_1 = fundamental frequency in megacycles
t = thickness in inches
k = a constant (½ velocity of sound in material measured)

A comparison of plate thickness measurements made over "m" months will show the average rate of corrosion attack on the surface opposite the prepared audigauge baseline.

$$(3)\ i = 12/m\ (t_o - t_m) = 12/m\left(\frac{.116}{fn_o/n} - \frac{.116}{fn_m/n}\right)$$

Where i = inches penetration per year
t_o = thickness original measurement
t_m = thickness after "m" months
fn_o = resonance frequency—original
fn_m = resonance frequency—after "m" months
n = harmonic

An example of the calculations for penetration rate at the test station are shown:

$$(4)\ i = 12/16\left(\frac{.116}{1.75/8}-\frac{.116}{1.78/8}\right) = .75\ (.530-.521) = .00675 \text{ inch/year}$$

Where k = .116
fn_o = 1.75
fn_m = 1.78
n = 8
m = 16

The results of an audigauge survey made in a clean products service T-2 type tanker are shown in Table 2. These measurements are made on similar members in the cargo tanks under the various operating conditions as classified in Table 2.

The results indicate that the average corrosion rate in the ballasted tanks has been reduced by 86.5 percent with cathodic protection.

Plaster Cast

The progress of attack in individual pits on bottom plates in crude service may be shown by the comparison of plaster contours (gypsum cement such as Hydrostone or Castone) made on annual resurveys (Figure 6). The pits must be thoroughly cleaned of corrosion products and the plaster must be applied with care to prevent entrapment of air. Two Monel pins welded to the shell provide a baseline for locating the test section in the vertical plane and for measurement (see arrow) of the progress of the pit. In unprotected tanks, the cleaned pits become active. A calcareous deposit and/or high pH is found in the pits if adequate cathodic protection is applied.

pH of Products in Pits

The author[6] has shown that decreasing pH of products in pits indicates increasing rates of pit penetration. For example, pit product pH 2.0 indicates a pitting rate of .100 inch to .150 inch/year. The deposition of calcareous salts or a pH above 9.0 in formerly active pits indicates that the corrosion rate is virtually zero.

Rust Scale

Adequate cathodic protection will loosen the rust scale from a tank. Once the rust scale has been removed, the absence of further rust scale formation—to be loosened or to be removed—indicates effective corrosion control.

Economics

The cost of cathodically protecting tanks varies widely. Two examples are used to illustrate the wide range in costs of protection/sq ft/year to obtain more than an 80 percent reduction in the corrosion rate.

EXAMPLE 1. A Middle East sour crude tanker with 12 day ballast periods—ballasted 15 percent of the time.
Cost of anode material and installation/100 sq ft of surface.....$12.00
Life of installation.....6 years

$$\text{Cost/sq ft/year} = \frac{12.00}{6 \times 100} = \$.02$$

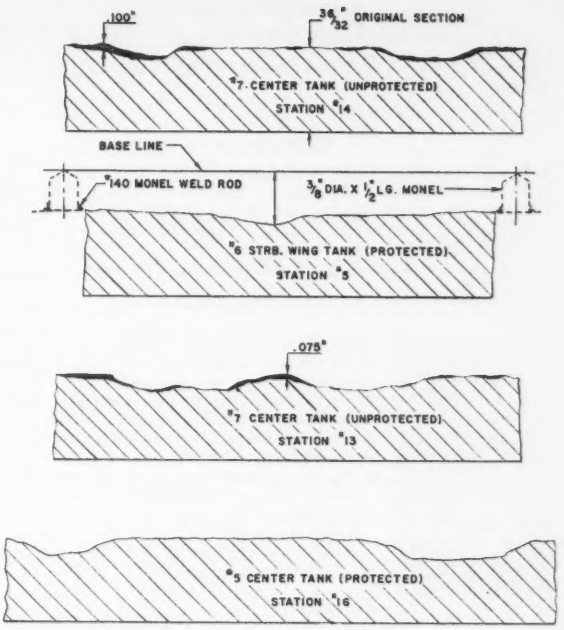

Figure 6—Shell plating profiles showing net change in pits between April, 1954 and March, 1955.

EXAMPLE 2. A tank used solely for ballast (with two day ballast periods and ballasted 40 percent of the time).
Cost of anode material and installation/100 sq ft of surface.....$20.00
Life of installation.....2 years

$$\text{Cost/sq ft/year} = \frac{20.00}{2 \times 100} = \$.10$$

The annual cost of protection may be compared with the cost of repair in an unprotected ballast tank.

Example: It is required that a bulkhead be repaired when the thickness is reduced by 25 percent.

Assume: The corrosion rate is .020 inch/year (both sides). A .500 inch plate weighs 20 lbs/sq ft and at $.50/lb, the replacement cost is $10.00/sq ft.

The bulkhead must be replaced in $\frac{.500'' \times .25}{.020''} = 6.25$ years.

The cost of corrosion is $\frac{\$10.00}{6.25} = \$1.60$/year/sq ft.

If the corrosion rate is reduced to .005 inch/year, repairs are not required for $\frac{.500'' \times .25}{.005} = 25$ years.

Conclusions

Cathodic protection has come of age in the past 10 years as a method of corrosion control on internal surfaces of ships.

The requirements for effective protection are:

1. The tanks must be ballasted with sea water at least 10 percent of the time.
2. Adequate protective current must be applied.

It should be pointed out also that the ballast procedure should be coordinated with the protective system to get the greatest yield in protective effect.

Acknowledgment

The author acknowledges the assistance of W. M. Laird and R. J. Walton in securing some of the data shown.

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DISCUSSION

Question by Charles W. Ambler, East St. Louis, Illinois:

You showed the magnesium anodes on straps which place them 15 inches above the steel bottom. Can this give protection from water which will settle out of crude or products in these tanks?

Reply by L. P. Sudrabin:

There is no protective effect on the tank bottom from magnesium anodes located above water which has settled from crude or product cargoes. In most cases, adequate residual protective effect to control corrosion is obtained from the alkaline calcareous deposits formed in the pits and on the active areas during the sea water ballast periods.

Any discussions of this article not published above will appear in the December, 1957 issue.

TECHNICAL PAPERS ON CORROSION WELCOMED

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

Progress in Prevention of Corrosion in Naval Aircraft*

By S. L. CHISHOLM and N. N. RUDD

Introduction

IN 1951 a paper was presented at the Detroit meeting of NACE[1] which was a most comprehensive discussion of the problems, the purposes, and the means employed to control and minimize the corrosion of naval aircraft. Since that time there have been developed and applied additional specific improvements in materials and methods. The following discussion is presented to supplement the previous paper, and as such is appropriately confined to changes occurring since 1951 which appear to be of significant general interest.

It is customary to classify and consider separately those means employed for the "permanent" protection of aircraft during an operational tour of duty, as opposed to the additional or supplementary temporary protection or preservation employed during periods of idleness, low maintenance, storage or shipment. Significant advances have been made during the past five years in both categories; both approaches are included in the present discussion.

Application of Navy Wash Primer to Aircraft

During World War II, Whiting and Wangner[2] of the Bakelite Corporation, under Navy auspices, empirically developed compositions known as wash primers or pretreatment coatings. One of these compositions was prepared by adding dilute alcoholic phosphoric acid to an alcoholic dispersion of zinc tetroxychromate in polyvinyl butyral. It yielded a primer having an unusually high order of adhesion to a variety of metals. Furthermore, paint films adhered well to it and the system so produced exhibited remarkable protection of the metal against corrosion. A specific composition[3] designated WP-1 has been used increasingly by the Navy for the treatment of bare metal surfaces prior to painting. The treatment of metal surfaces by this wash primer is considered one of the most important steps in corrosion prevention in recent years.

Several authors[4,5,6] have speculated on the mechanism by which this wash primer acts to provide its protective and adhesive action. Another author[7] has reported an initial attempt to investigate the structure of this wash primer film on an iron or steel surface, its mechanism of formation, and its mode of action in retarding corrosion and increasing paint adhesion. The results obtained indicated that the wash primer treatment on iron surfaces: (1) generates a protective oxide film, (2) supplies chromate for repair of any damage to such a film, (3) supplies a zinc phosphate analagous to the films produced in phosphate treatments, and (4) superimposes on these a polyvinyl butyral film which acts as a good mechanical protection and as a bond for subsequent

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Abstract

Most naval aircraft, because of the corrosive environment in which they are employed, require the best available surface protection. Thin resin and lacquer films have exhibited good metal adherence and excellent resistance of protected parts to marine weathering. Many magnesium alloy components are effectively protected by high bake silicone resin formulations. Pigmented silicone resin coatings on engine cases and numerous other cast magnesium aircraft components, have been in operational use for about three years with good results. Use of such bake application methods is necessarily limited to certain aircraft parts.

Protection of naval aircraft parts during standby and storage periods is now being accomplished by two new approaches, (1) thixotropic preservation formulations (gel structures) and (2) vapor corrosion inhibitors. A group of 22 North Island aircraft engines were carefully preconditioned, ten protected by conventional oil mixtures and twelve by a vapor corrosion inhibitor. The test engines were sent to exposure areas ranging from arctic to tropic conditions. After only six months, the engines protected by oil mixtures showed serious cylinder wall corrosion while those protected by the vapor corrosion inhibitor were still in acceptable condition after 18 months. 8.9.1

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paint films. Further investigations were reported as in prospect or progressing, including studies of application to magnesium and aluminum.

Just as actual production application to ship's steel surfaces quickly followed the early empirical testing, so also by late 1950 and early 1951 attempts were underway by the Navy Bureau of Aeronautics to utilize the wash primer technique on aircraft aluminum exterior painted surfaces. By late 1951 production utilization of the technique was well advanced. The standard exterior paint finish for aircraft was at that time one coat of zinc chromate primer and two top coats of pigmented nitrocellulose lacquer. This was, accordingly, preceded by the wash coat primer application following the initial specified surface treatment of the bare aluminum alloy, such as anodic treatment for seaplane hulls, or the permitted alternate treatments[8] on other exterior surfaces. With the release of subsequent modified painting specification[9] an alternate wash primer[10] also was provided to permit a smoother over-all aerodynamic finish for high speed aircraft, and application instructions were finalized in 1953.[11]

The foregoing development and additional application of wash primer has very greatly improved the protection of the exteriors of naval aircraft when used in conjunction with the standard nitrocellulose lacquer finishing materials. This is particularly gratifying because of the development of the operational need for large seaplanes, often moored in the sea when not in flight, and the utilization of higher strength light alloys requiring improved protection. Used on the exterior of carrier and other landplanes, it also has greatly decreased the demands of operational paint maintenance.

Protection of Bare Alclad Aircraft

Most naval aircraft, because of the corrosive environment in which they are employed and because of the exigencies of military deployment and utilization, require the best available paint protection. Some non-combat aircraft, however, such as transport or utility aircraft operate from equipped fixed bases and over scheduled routes, in a manner similar to that of civil air lines, and are basically non-military. Such aircraft, like civil air transports, frequently have unpainted alclad exterior surfaces which are subject, particularly with the exigencies of cleaning and brightening maintenance while in military operations, to superficial corrosion in marine atmospheres. These surfaces have posed a problem at the time of major overhaul because of the excessive effort required to restore them to a like-new condition, and because of objection to the resultant removal of a significant or excessive amount of the protective cladding.

It is practicable to remove or inhibit residual corrosion by chemical treatments, but not to restore the original appearance if the corrosion has progressed to a significant extent. Painted-on insignia, squadron and other markings, when removed, also leave shaded markings on the oxidized skin.

Analysis of the factors involved appeared to indicate that only a moderate increase in protection was required to prevent either the initial or progressive deterioration of the aluminum cladding, and that only a moderate obscuring action was required to restore a satisfactory surface appearance. Previous experience with methacrylate resin films had indicated that very thin films exhibited excellent adhesion to aluminum and that such thin films exhibited a particularly good adhesive strength to cohesive strength ratio. It was found practicable to apply a thin coating of a semi-aluminized butyl methacrylate lacquer[12] which shows excellent adhesion to such surfaces after pretreatment with a detergent alcoholic phosphoric acid wash (or a similar surface treatment), good mechanical and weather resistance and otherwise very adequate performance. The use of a minimum of aluminum pigment paste (2 ounces/gallon) permits a close approximation to the appearance of any unpainted aircraft on the same flight line but adequately covers or obscures surface blemishes and discontinuities.

Because of its excellent performance when used on aircraft under overhaul, further application of methacrylate resin films has been extended to new alclad aircraft before delivery for operational use, in order to minimize subsequent maintenance. This product gives a protective film of the order of 0.2 mil thick. Breaks or abrasions in the film, if they occur, have therefore little adverse aerodynamic drag even on the leading edges of fast aircraft. This material has, accordingly, also been employed successfully on trainer jet aircraft with clad skins to reduce maintenance effort and service deterioration.

Improved Protection of Magnesium Components

For many years the only major uses of magnesium alloys on naval aircraft were in engine cases and in aircraft wheels. The first were bathed internally by lubricating oil, were protected externally by baked enamel coatings, and generally posed no particularly severe corrosion problems.

Deterioration of magnesium wheels in naval aircraft was somewhat more of a problem. In more recent years the utilization of magnesium alloys in aircraft has very greatly increased, both in structural sheet form and in a wide variety of cast and forged parts. With gas turbine engines, also, the differentiation between exterior and interior surfaces tend to disappear. When jet engines or magnesium parts are employed on seaplanes, amphibians or helicopters engaged in sea rescue work the corrosion problem may become really acute. Tail gun turrets employing operational magnesium parts can, for example, become quite inoperative after a very few sea landings and take-offs, when protected only with conventional protective paint finishes.

Bake Cured Phenolic Resin Coatings

During World War II there was developed a bake cured phenolic resin coating[13,14] for the surfaces of the internal steel parts of reciprocating aircraft engines. Subsequent to the war this material found numerous industrial uses. During the early post-war years the practice also developed of employing this phenolic resin as an interior coating on magnesium

engine and carburetor cases, and the like, and as a prime coat under the baked-on exterior enamel finishes. For these purposes it proved superior to previously used materials, such as the air drying zinc chromate primer previously used under the baked enamel top coats. This was due in part to its high dielectric characteristics and in part to its baking compatibility.

The use of the phenolic lacquer on magnesium cases and other parts had, however, two distinct limitations. First, it was somewhat brittle, and chipped or marred too readily, particularly at sharp edges or discontinuities. Secondly, and even more serious, the phenolic film was alkali sensitive, and the bond to magnesium metal was seriously impaired by the alkaline reaction of any magnesium hydroxide resulting from the incipient corrosion of the magnesium metal. It was found that any break in the finish frequently resulted in progressive flaking off of the paint and progressive corrosion of the magnesium part. Based on these observations a search was made for substitute materials less brittle, more tough, and also more adequately alkali resistant. Such materials were found to be available in the high bake silicone resin formulations offered as recent paint developments.

Pigmented Silicone Resin Coatings

Pigmented silicone resin coatings on engine cases and numerous other cast magnesium metal aircraft components have been in operational use for about three years with very gratifying experimental and operational results. In most instances this particular finish was initially applied to seaplane components to correct early failure of the conventional protection and to prevent further deterioration of the part. In practically all cases observation after an operating tour of 14 to 16 months disclosed a condition with no discernible deterioration or change. This development is sufficiently recent that no military specifications other than local directives have yet been issued to control the procurement or use of the silicone resin finish. This finish is currently being employed both as a single coat interior finish and as a prime coat under baked enamel top coats for exteriors.

Use of baked silicone finish is necessarily restricted to those parts which can be bake-cured and it cannot be used on the external or structural magnesium sheet or sections as used in assembled aircraft structures. For the latter use at this time it is only possible to report that considerable effort is being expended in the testing and development of better air drying or curing inhibitive primer formulations and top coating materials. The most promising primers for magnesium presently combine two or even three combined inhibitive pigments and the more promising top coating materials presently include polyester resin and polyester-epoxy resin formulations. Fairly rapid and successful accomplishment in this field is confidently anticipated, but more detailed discussion would be premature at this time.

Development of Thixotropic Reciprocating Engine Preservative Compound

The materials and techniques covered to this point involve permanent protective schemes. In addition to the necessity for protecting exposed aircraft surfaces during operational service, there is the very real problem of providing interim protection to working parts and installed accessories during periods of inactivity ranging from the week-end shutdown to extended pool storage. This is usually accomplished by means of temporary films, inhibited oils, soft film greases, or dry film residues, of which a great variety of materials are available.

Two new approaches to interim protection problems have developed sufficiently in Navy application since 1951 to warrant special comment: (1) the development of a thixotropic preservative formulation for the protection of inaccessible surfaces, particularly the internal cavities of reciprocating aircraft engines, and (2) the increasing use of vapor corrosion inhibitors for the protection of all types of aeronautical materials.

The initiation of the use of thixotropic or gel structure engine preservative compound[15] was the culmination of a series of developments on engine preservation which began in 1934 with the formulation by the Ethyl Gasoline Corporation of a special preservative compound. This compound consisited of acid lard oil containing aluminum stearate and triethanolamine, together with butyl alcohol as a mutual solubilizing agent. This early preservative proved unsatisfactory for aeronautical engine use and was shortly superseded by engine oil containing triethanolamine and triethanolamine oleate. The use of such a highly hygroscopic amine for the neutralization of lead halide residues (from leaded fuel) also proved unsatisfactory and actually deleterious to engine interiors, and in 1938 a change was made to a mixture of a tertiary alkylated phenolic amine (Hyamine B) and an appropriate grade of paralketone in engine oil. Paralketone (NAVAER RM-61) was a mixture of high molecular weight paraffin alcohols, ketones, esters, and acids produced by the partially controlled oxidation of paraffin waxes.

About 1940 a further change was made to engine oil containing dissolved petroleum sulfonates, plus an oil soluble amine with a specified hydrobromic acid neutralization requirement (AN-VV-C-576). About the same time measures were instituted to supplement the protection offered by internal preservative films with dehydration techniques, employing silica gel plugs in spark plug and drain plug openings and enclosing the complete engine assembly in a sealed pliofilm envelope containing additional cloth bags of desiccant.

During World War II pliofilm bags deteriorated rapidly under tropical storage, with the result that the Bureau of Aeronautics initiated and developed the use of cylindrical steel engine containers, hermetically sealed and pressurized. The combination of pressurized dehydration, together with the use of a petroleum sulfonate containing oil preservative during the preservation run-in of engines has given excellent results. Engines so preserved have con-

sistently shown no significant deterioration over periods of ten years or more. While preservative materials now in use have changed,[16] the basic procedure is still the Navy standard for stored uninstalled engines.

This method, however, is not applicable to the very numerous reciprocating engines installed in aircraft during periods of idleness incident to shipment or storage, nor to engines long removed from containers or mounted on air shipment stands. Maintaining the necessary supplementary dehydration and preventing moisture condensation within such engines proved to be an extremely difficult condition to effect, since the engine itself is not a hermetic enclosure.

Early dehydrator plugs were made of plastic with a measurable moisture permeability. Substitution of glass plugs involved problems of effective gasketing. Even with the best plug design, and elaborate sealing of engine openings, moisture still collected inside engine cavities, and caused serious damage before it could diffuse to and be removed by any internal desiccant provided. The particular need for dehydration is occasioned by the fact that even effective polar preservative oil will drain continually and indefinitely from vertical surfaces until too thin for protection against condensed moisture. Moisture droplets will also in time penetrate and accumulate below even thick or viscous horizontal oil films.

As a result of the limited success in protecting installed engines a new type of preservative was initially specified and procured in 1944 and first put to use as early as 1945. This material, MIL-C-5545, is compounded from heavy engine oil, petroleum sulfonates and an acid neutralizing amine, plus a suitable proportion of petroleum microcrystalline wax. When this preservative oil mixture is cooled through the transition temperature of about 120 F, the latter constituent causes its coalescence to a gel structure with practically a nil draining rate, but which under shear movement reverts to a liquid.

The field use of this new formulation was delayed by the slow development of application equipment and techniques. Operating activities hesitated to use such a heavy base material for internal engine protection because of the danger of engine damage during depreservation. Only in the past two or three years has the real potential of this material become evident. However, use of heavy duty MIL-C-5545, with modified hot application techniques,[17] is now the accepted Navy procedure for the internal protection of installed reciprocating aircraft engines for any storage period exceeding 60 days. The engine is given a preservation run on a 50-50 mixture of the preservative and operating oil and an injection of the same mixture through the intake system at time of shutdown, followed by a fog spray of the cylinder walls through the spark plug openings while the engine is still warm. No special sealing of the engine is practiced and dehydration as such has been discontinued. Results have been highly satisfactory and the life of such protection applied to a rust-free engine is still being extended.

A series of field tests were conducted in connection with the evaluation of vapor corrosion inhibitors as an engine preservative in which the control engines were given a MIL-C-5545 treatment. The test engines were stored, with no supplementary protection except wooden engine boxes for handling purposes, both at North Island and under extreme exposure conditions at several overseas locations, for periods up to three years. While in several instances the wooden engine container was so severely deteriorated as to make it unsafe for handling, the control engines at teardown and examination were still free from serious attack and comparable to their condition at time of original preservation just out of major overhaul. As a result of these and similar tests, the life of installed engine preservation using MIL-C-5545 materials has been extended successively from 30 days to 120 days, to one year and presently to two years, with no preservation renewal required.

During this same test series, it was further demonstrated that a spray coat of MIL-C-5545 material gave excellent protection to external engine surfaces if the coating was provided a minimum of protection against direct washing by rain or salt spray. However, in those areas not sheltered by engine cowling, inhibited grease type films, such as Specification AN-C-124 material[18] were more tenacious than MIL-C-5545 and showed better weathering characteristics than conventional heavy duty hard film preservatives[19] for extended storage periods under severe exposure conditions.

The Advent of Vapor Phase Corrosion Inhibitors

During World War II there appeared in the Chemical Section of the Navy Bureau of Aeronautics a representative of the Shell Chemical Company bearing a white crystalline sample described as a vapor phase corrosion inhibitor with prospective uses for protecting ferrous alloys during storage and shipment. Because information as to its composition was not immediately available the sample was forwarded to the Chemical Section of the Naval Research Laboratory for identification. Within two weeks that group identified the material as diisopropyl ammonium nitrite and had synthesized a duplicate sample.

Vapor phase corrosion inhibitors were not exactly new, volatile amines such as morpholine having been used in vapor degreasing solvents and in steam boiler systems previously to prevent or reduce the corrosion characteristics of vapor condensates. This kind of material and its prospective uses were however distinctly new. It represented a stabilized substituted ammonium nitrite and a whole new class of solid chemical compounds with a significant and controlled vapor pressure, which in addition possessed corrosion inhibitive characteristics. Such materials are stable chemical salts formed from the combination of a volatile acid and volatile base, and thus themselves possess a measurable vapor pressure. When these compounds are introduced in sufficient quantity within any enclosure, they tend to saturate the enclosed atmosphere with vapors and presumably also deposit polar surface films throughout. In the case of the substituted ammonium nitrite, such action should be and actually is, adequate to prevent the corrosion of ferritic steels against the attack of

enclosed or admitted atmospheric moisture which might condense and act on such surfaces, so long as a reasonable excess of the corrosion inhibitor remains or persists therein. Its effectiveness is indicated by the fact that as little as 0.02 percent dissolved in water prevents the corrosion of immersed steel.

The persistence characteristics of such compounds within a typical but nonhermetic enclosure, apart from possible chemical instability in the environment, together with the rate of diffusion and distribution within the enclosure depends on the vapor pressure of the salt used. The vapor pressure may vary considerably between salts, but is ordinarily low. The mechanism of vaporization of the salt appears to involve its dissociation into acid and base and an equilibrium recombination within and on surfaces in the enclosure. If there is a wide difference in the volatilities of the acid and base formed, as for example in the case of ammonium benzoate, undesirable effects may result. The more volatile constituent, ammonia, will tend to escape much faster, leaving an acid residue. In the case of ammonium benzoate, an accumulation of about 2 percent free acid makes the material ineffective in preventing the corrosion of steel. Thus good chemical stability depends on a reasonable balance of volatility as between acid and base. This balance holds sufficiently in the case of certain amine nitrites, including both diisopropyl and dicyclohexyl derivatives.

Other environmental hazards to good stability occur in the presence of extraneous chemical reactants, such as those resulting from hydrolysis of lead halide deposits within aircraft or other engines run on leaded fuels, which in turn, produce volatile halogen acids. Such hazards can, and are being reduced in newer formulations by the use of a volatile neutralizing additive, designed to prevent decomposition and consequent early loss of the corrosion inhibitor and its protection. When such a breakdown does occur, it is usually in severely localized areas within the enclosure. The same technique might obviously be used to improve the performance of a non-ideal vapor phase corrosion inhibitor, such as ammonium benzoate, by the addition of such materials as ammonium carbonate.

Further discussion of the theoretical factors to be considered in developing and using vapor corrosion inhibitors will not be attempted at this time. The type of chemistry involved in their activity is fairly obvious, and the characteristics of particular compounds are covered in published literature. The case for the Shell Oil Company products has been well documented.[20,21,22,23] Several competitive materials have appeared on the market, some based on older British and European formulations. Rock Island Arsenal prepared an extensive general bibliography in 1953 covering all published information on vapor corrosion inhibitors available up to that time.[24] Considerable further information has been published subsequently.[25,26]

The use of vapor corrosion inhibitors offers a new approach to the control of corrosion problems. While the introduction of such materials to the preservation field is at least ten years old, much still remains to be done. Areas are yet to be developed where the method is technologically sound and economically justified, and it is yet to be determined to what extent the use of such materials can and should replace or supplement previously recognized methods, such as the use of polar slushing compounds, dehydration techniques and the like. While an increasing interest in the potential of these materials for the protection of aeronautical equipment is being evidenced, many of the present applications are being conducted on a field test basis and the controlling military specifications for the procurement[27] and use[28] of vapor corrosion inhibitors are still in the process of evolution.

Specification materials presently available are limited to impregnated or coated wrapping papers or lined envelopes although test applications and controlled production use of both powder injection and solvent spray applications have and are being used with excellent results. As an indication of the possibilities of the use of VCI materials in corrosion control, a brief summary of two test series follows, one application using impregnated paper as the VCI carrier, and the second employing direct VCI powder injection.

Protection of Naval Ordnance Using Vapor Corrosion Inhibitors

The protection of naval ordnance was an obvious early application for VCI materials, both because of the almost exclusive use of corrodible steels in its construction and the problems involved in providing adequate storage protection, with a minimum of maintenance and depreservation required to make it ready for use. Most war veterans remember all too well the work involved in disembalming newly issued automatic pistols or rifles protected with cosmoline and similar grease-like coatings. It was in connection with ordnance equipment that the only practical use of VCI materials prior to VJ day was reported (i.e., the protection of shell case cavities between foundry and filling station.) If shell cases were rusty on arrival, they required derusting. If they were coated with a preservative, this also required careful removal. It was found that a kraft paper insert impregnated with a VCI material protected shell cavities adequately and merely required removal of the inserts before loading the cases.

Naval aeronautical ordnance in 1945 consisted chiefly of machine guns and 50 mm cannon. Late that year a test was initiated at Naval Air Station, North Island, to determine the applicability of VCI techniques to long-term storage protection of overhauled guns and cannon.

During overhaul guns are disassembled, each part wet abrasive blasted, again parcolubrized, assembled, lubricated, test fired and preserved. The customary preservation for extended storage at that time was basically a dip coating of hard film preservative, (AN-C-52). However, the use of such a material had several objections. Application to certain operating

mechanisms was either undesirable or impracticable, and experience had demonstrated that after a lapse of time the preservative coating hardened sufficiently so that guns and cannon so preserved required considerable rework before they could again be made operable. The hardened coatings could not be readily removed in whole or in part with the facilities available even at a Class A shore station.

Shipment of embalmed guns to fleet activities entailed even more difficulties, and such shipments were involved in the delivery of combat aircraft for immediate use. An obvious need existed for a method of preservation which gave 100 percent protection to guns lubricated only with their operational lubricant, and which could be as easily removed as a wrapping from a package leaving the gun in a ready-to-fire condition. The application of VCI materials was visualized as answering all of these requirements.

The experimental method adopted was to spiral wrap the gun or cannon assembly with a continuous strip of kraft paper impregnated with VCI, insert a similar spiral in the bore, enclose the whole in a stockinet bag, and hot dip the package in plastic sealing compound.[29] The only supplementary protection applied was a dip coat in the normal operational lubricant before wrapping. The bulk of the test guns were shipped to an ordnance storage activity, but a number were retained aboard the Naval Air Station, North Island for subsequent periodic evaluations. The final retained samples were opened after ten years of outdoor boxed storage.

The results were extremely interesting. In all cases examination showed no serious visual deterioration and all guns operated satisfactorily on test firing. It was also of interest to note that the hot plastic dipping apparently volatilized some of the corrosion inhibitor from the paper wrap and distributed and redeposited it on and within the gun, showing in some areas temperature gradient patterns of deposited crystalline VCI. This residual material was not removed in subsequent handling and testing, and apparently had no adverse effect on the operation of the equipment.

In local tests the plastic overcoat used was an oil-ethyl cellulose mixture, which subsequently showed superficial surface cracking and weathering when directly exposed to actinic rays. Poor low temperature characteristics on handling were reported unofficially from other colder storage points, and as a result plastic overcoating was never used except on a test basis. However, current procedures for uninstalled ordnance preservation call for a VCI overwrap further protected by a floating bag type vapor barrier envelope, with the gun predipped in light preservative oil and mounted in its container. While the results of this modification are not available over as long a storage period as in the original test, the degree of protection being obtained is completely adequate to date. Furthermore the general conclusion to be drawn from the earlier test results appears inescapable, that since no deterioration of protection occurred during a ten-year interval, this basic method is one that can give complete protection to steel mechanisms and parts for an indefinite period with zero preservation maintenance. This factor is of prime significance to the military who normally between periods of active hostilities have large amounts of material to store and maintain in such a manner that it will be immediately available for use when needed.

VCI Protection of Reciprocating Aircraft Engines

The final test program, to be summarized, involves the use of powder injection of Shell Chemical's VPI-260 for the internal protection of reciprocating aircraft engines. Engine protection has always been a major military problem as indicated in the discussion of the development of a thixotropic film preservative. When using conventional preservatives an engine run-up is essential if complete internal protective coatings are to be obtained. Any disturbance of a protective film requires a second preservation run for its restoration. Special equipment is needed to apply hot spray cylinder wall coatings in sufficient quantity to assure protection but at the same time prevent excessive application and resulting hydraulicing of cylinders when the engine is depreserved. The human element is ever present in assuring that 100 percent cylinder wall coverage is obtained. Engine preservation procedures are time consuming, and the more extensive the protection applied, the greater detail required in its removal.

The possible use of VCI materials for engine protection was one of the earliest considerations for these materials, both by the Military and by commercial airlines. The first Navy field tests were initiated at North Island in 1951 when a group of 22 aircraft engines were overhauled and their condition catalogued expressly for test purposes. Control engines were protected with heavy type MIL-C-5545 material, a second group of engines were treated with conventional inhibited oil mixtures (AN-VV-C-576) and dehydrating agent, and twelve engines were protected only by injection of VPI-260 powder into engine cavities after the completion of the acceptance run on operating oil. Approximately 75 grams of VPI powder was used in each engine, applied through spark plug openings, sump standpipes and accessory covers in such locations as to reach each major internal engine cavity.

The test engines, with no protection other than a wooden box for handling purposes, were distributed to exposure areas ranging from arctic to tropic conditions, with ten engines being held in the San Diego area with box tops removed and the engines exposed directly to the weather. Engines protected by conventional methods with inhibited oil mixtures, supplemented by dehydration techniques used for installed engines, showed serious cylinder wall corrosion within six months and were removed from test. The first VPI protected engines and one control engine were returned from the Philippine area after 18 months of unprotected storage. While the engine boxes were badly deteriorated, engines preserved with both VPI powder and MIL-C-5545 were still

in acceptable condition. The scattered attack in the VPI treated engines occurred for the most part under sludge deposits and in dead-end oil passages inaccessible to VPI vapors.

Subsequent withdrawals from test and examination were conducted on groups of engines after two-year and three-year storage periods. Four of the VPI protected engines were given test stand runs with no depreservation after their return from storage areas, with performance ratings comparable to their original condition at time of overhaul. Not until three years of severe exposure was there a noticeable difference between the control engines and the VPI injected engines. Cylinder walls, the most critical engine area, began to show slight to moderate attack in the final VPI engine examined.

This check of the effectiveness of vapor corrosion inhibitors in protecting major assemblies was a particularly severe test. Not only were exposure conditions unusually severe, but also not even an inhibited light oil was used to provide interim protection during the period necessary for the vaporization and distribution of the vapor phase inhibitor. No supplementary protection was applied to hard-to-reach or inaccessible passages. The vapor inhibitor, present originally in small amounts, was undoubtedly further reduced in effectiveness by the presence of oil pools and operating oil coatings on all surfaces. No additive was present to counteract the effects of lead salts, hydrobromic acid or other combustion products present in any internal combustion engine after operation on leaded fuel. A variety of non-ferrous metals and alloys are contained in an aircraft engine, many of which have a laboratory history of staining and surface attack in the presence of dicyclohexylammonium nitrate. No such staining was noted in any of the test engines.

As a result of the above tests, an additional preservation approach to engine protection is gradually being adopted by naval activities. The use of VCI materials offers a positive method, easy to apply, for the protection of inoperable engines. When thixotropic preservative films in operable engines are disturbed during incorporation of changes, the use of VCI injection makes possible the represervation of such engines without a test stand run. As a supplement to MIL-C-5545 protection, VCI can be injected into cylinders with no inspection required to assure the absence of "holidays" in the preservative film, and with no danger of cylinder hydraulicing at time of depreservation. Present policies restrict VCI engine protection to one year storage with one represervation permitted. However, this one year limitation may well be further extended with the use of newer type VCI materials containing hydrobromic acid neutralizing additives and a small percentage of more volatile compounds to give early protection, supplemented by an engine run-up on an inhibited oil in order to reach inaccessible passages.

The broader implications for the ultimate use of vapor corrosion inhibitors in the protection of materials against corrosive attack are far-reaching. Definite limitations are involved, but in approaching any corrosion problem consideration should be given to the possibility of employing some type of vapor phase material in its solution. This is definitely a new and only partially explored preservation concept.

Acknowledgments

From among the many participants in the developments under discussion, appreciation is expressed to Carl Becker of Philadelphia for helpful suggestions in the initial formulation of MIL-C-5545 and to Harold Acker of Los Angeles for similar assistance with MIL-L-8641. Within the naval service, major credit is due Nate Promisel, Francis Somers, Albert Malloy and Jack Witten of the Bureau of Aeronautics. Lt. J. H. James also developed the first successful application technique for MIL-C-5545. Other participants are literally too numerous to mention specifically and include both those in the naval service and industrial representatives whose contributions and participation have been most helpful and essential.

NOTE: The opinions and conclusions expressed herein are those of the authors and do not necessarily represent the views of the Department of the Navy or of the Naval Establishment.

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Any discussions of this article not published above will appear in the December, 1957 issue.

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The Performance of Alcan 65S-T6 Aluminum Alloy Embedded in Certain Woods Under Marine Conditions*

By T. E. WRIGHT,[1] H. P. GODARD[1] and I. H. JENKS[2]

Abstract

Laboratory and marine exposure tests were carried out to assess the performance of Alcan 65S-T6 (6061-T6) aluminum alloy embedded in certain woods under marine exposure conditions. The tests also involved the assessment of several protective paint systems designed to protect the wood in the simulated hull sections used in the tests.

The relative corrosivity to aluminum of the woods tested under ship hull conditions is: rock elm (least), mahogany and British Columbia fir (most). Only very slight corrosion occurs when the aluminum is coated with zinc chromate priming paint even in the most corrosive woods after extended immersion periods in sea water. Alcan 65S-T6 (6061-T6) has a corrosion resistance greatly superior to that of Alcan 24S-T3 & 4 (2024-T3 & 4) under the same marine exposure conditions. Anodizing increases the corrosion resistance of both alloys, particularly if this treatment is followed by a coat of zinc chromate primer. The use of wooden dowels to protect aluminum embedded in these woods is also beneficial. Among the variations in paint systems which were tested, the most complete paint system, comprised of sealer + barrier layer + copper antifouling paint provided maximum protection from corrosion. Where pitting occurred on 65S-T6 (6061-T6) embedded in sea water wetted wood, it has been shown that the rate of penetration falls off sharply with time. **6.4.2**

Introduction

ALUMINUM IN SUCH forms as plate, sheet, foil, extruded sections and tubing, castings and forgings, rivets and bolts, is being used in increasingly large quantities in a wide variety of applications in shipbuilding. Indeed, it may be said that as a result of recent technical developments and experience over the past fifty years, aluminum alloys are regarded today as one of the most significant and promising classes of shipbuilding materials. Their use in shipbuilding has been approved officially by Lloyds register of shipping and their "Tentative Requirements for Quality and Testing of Aluminum Alloys for Shipbuilding Purposes" makes recommendations for the use of aluminum alloys in the structural assemblies of large vessels. Moreover, the Naval authorities of many countries have accepted aluminum for naval construction.

Some six years ago the Naval authorities in Canada became interested in building a number of craft using Alcan 65S-T6 (6061-T6)[3] aluminum alloy bolts to fasten wooden hull planking to aluminum structural ribs. At this time it was necessary to determine on a fairly urgent basis the corrosion behavior of aluminum bolts embedded in wood in contact with sea water. As a result a program to acquire this information was instituted by Aluminium Laboratories Limited, Kingston.

Description of Materials

Four woods supplied by the Davie Shipbuilding Company, Lauzon, Quebec, from stocks to be used in building ships were investigated (viz: mahogany, Canadian rock elm, British Columbia fir, and gum wood). These woods were supplied in the form of dry planks 2 inches thick (some elm planks were 4 inches thick), and varying in width from 6 to 10 inches.

The composition and description of the aluminum alloys from which the specimens were taken are shown in Table 1. Alcan 24S-T3 & 4 (2024-T3 & 4) was included for comparison purposes; galvanized steel bolts with zinc coating of 2-oz sq ft, similar to those in 65S-T6, (6061-T6) obtained from the Steel Company of Canada, also were exposed for comparison purposes. In some tests sheet zinc (composition: Cu 0.07 percent, Fe 0.01 percent, Zn by diff. 99.92 percent) coupons of 0.090 inch thickness were exposed as controls.

Short Term Experiments

Aqueous Wood Extract Tests

A weighed amount (104 grams)of sawdust made from planks of each type of wood was placed in a 2 liter beaker and covered with 1.5 liters of distilled water. The combined filtrates were boiled down to 100 mls and the pH determined. Weighed coupons (¾ inch x 3 inches) each of degreased 65S-T6 and 24S-T3 were placed in each extract and allowed to stand at room temperature for 30 days. In the case of the gum wood sawdust test, the immersion period was six days and a coupon of zinc sheet similar to the aluminum alloy coupons was used for control purposes.

A few white nodules of corrosion products formed on the 65S-T6 and 24S-T3 sheet coupon immersed in the mahogany and fir wood aqueous extracts. Shallow pits were found under the nodules. The elm wood extract was non-injurious to the aluminum and the gum wood extract caused uniform attack. Zinc,

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(3) The alloys used in these investigations are those of the Aluminum Company of Canada (e.g., Alcan 65S-T6). U. S. Aluminum Association equivalent is 6061-T6.

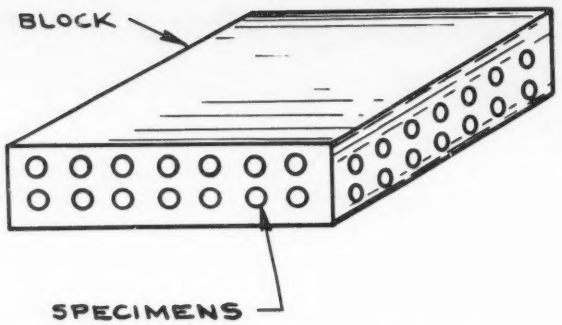

Figure 1—Small wooden block showing specimens inserted in block.

which was tested only in the gum wood extract was rapidly affected by general dissolution. The results of these tests are summarized in Table 2.

Sawdust Immersion Tests

A measured volume (approximately 750 cc) of sawdust from each wood was placed in crystallizing dishes. Degreased and weighed sheet specimens of 65S-T6, 24S-T3 and zinc (3 inches x 4 inches) were immersed in the sawdust for 30 days except for zinc specimens in sawdust from mahogany, Canadian rock elm and British Columbia fir, in which cases the test period was 14 days. The tests were run at room temperature. The sawdust was kept moistened with distilled water and evaporation was minimized by covering each dish with heavy aluminum foil. The results are summarized in Table 3.

Although in the case of immersion in sawdust from mahogany, fir and elm, the results were inconclusive, it was shown conclusively that 65S-T6 was superior in the gum wood sawdust. The 24S-T3 specimens in gum wood sawdust showed considerable pitting and exfoliation attack with some pits 26 mils deep after 30 days. The zinc specimens were uniformly corroded but not pitted.

Intermediate Tests

Humidity Cabinet Tests

Triplicate blocks of elm, fir and mahogany, measuring approximately 7 x 7 x 2 inches, were prepared. Aluminum alloy test specimens were prepared in the form of 65S-T6 and 24S-T3 ¼ inch extruded rods, 2 inches long. Some specimens were anodized in 15 percent sulfuric acid for 30 minutes at 21 C and a current density of 15 amps/sq ft. The anodized film was sealed in boiling water. The rod specimens were driven into pre-drilled holes in the various woods and half the holes were plugged with wooden dowels of ¼ inch diameter and ½ inch length.[4] The aluminum specimens protected by the dowels were given a coating of zinc chromate priming paint before they were driven into the wood. All holes were the same diameter as the specimens so that some pressure was required to drive them. The dowels were dipped in Jeffrey's Superseal Jointing Compound No. 241A.[5]

Five aluminum specimens were prepared for each different test, a total of 40 for each block. The specimens were driven into the sides and ends of the blocks in a uniform arrangement (see Figure 1). Nine blocks prepared in this way were placed in a humidity cabinet (120 F and 100 percent relative humidity with continuous condensation). After time periods of two, six and eight months, the specimens were removed from the wooden blocks, cleaned up, dried and weighed.

The corrosion rates are shown in Table 4. The humidity cabinet tests showed that 65S-T6 and 24S-T3 in fir, elm and mahogany were not appreciably attacked even though the wood remained damp and warm for a long time. The specimens in the elm wood were least affected, mahogany next and B. C. fir most affected. Alloy 65S-T6 was more corrosion resistant than 24S-T3. Corrosion was by pitting except for a few cases of exfoliation attack in 24S-T3 specimens. Anodizing was beneficial to the aluminum specimens. However at the conclusion of the test the benefits of anodizing were not as apparent, particularly for 65S-T6. Where pits occurred on the anodized specimens they were quite large although the unaffected areas appeared very bright.

TABLE 1—Composition and Description of the Alcan Aluminum Alloys Tested

Alcan Alloy* and U.S. Equivalent	Form	Cu%	Fe%	Mg%	Mn%	Si%	Ti%	Cr%
65S-T6 (6061-T6)	Sheet (0.064")	0.36	0.37	1.06	...	0.64	0.05	0.22
65S-T6 (6061-T6)	¼" rod	0.25	0.34	0.95	...	0.55	0.01	0.24
65S-T6 (6061-T6)	⅜" bolts	0.23	0.34	1.01	...	0.49	0.01	0.13
65S-T6 (6061-T6)	Sheet coupons (0.125")	0.29	0.40	0.94	...	0.57	...	0.20
24S-T3 (2024-T3)	Sheet (0.064")	4.52	0.34	1.44	0.63	0.25	0.01	...
24S-T3 (2024-T3)	¼" rod	4.64	0.27	1.52	0.64	0.20	0.01	...

* Hereafter referred to by Alcan designation.

[4] The grain of the dowels was parallel to the longitudinal axis and perpendicular to the surface of the block rather than parallel as would be the case in shipbuilding practice; dowels of this small size could not be prepared otherwise.

[5] Alfred Jeffrey & Company, Marshgate Lane, Stratford, London, England.

TABLE 2—Corrosion Rates in Aqueous Wood Extracts

ALLOY	Mahogany (pH 4.8)		Fir (pH 3.1)		Elm (pH 8.8)		Gum Wood (pH 4.3)	
	mpy*	Remarks	mpy*	Remarks	mpy*	Remarks	mpy*	Remarks
65S-T6	0.05	(Nodules)	0.55	(Nodules)	nil	No attack	4.9	General attack
24S-T3	0.06	(Nodules)	0.31	(Nodules)	nil	No attack	1.8	General attack
Zinc	...	...	...	...	...	...	6.5	Gassing of sample

* Calculated from the weight losses.

TABLE 3—Corrosion Rates in Moist Sawdust

Alcan Alloy	MILS PER YEAR			
	Mahogany	Fir	Elm	Gum Wood
65S-T6	0.25	0.36	0.12	1.6
24S-T3	0.18	0.44	0.09	3.8
Zinc	0.83	0.26	1.4	3.0

TABLE 4—Corrosion Rates in Woods in Humidity Cabinet

Wood	Alloy	Months	MILS PER YEAR			
			BARE		ANODIZED	
			No Dowels	Dowels and Paint	No Dowels	Dowels and Paint
Mahogany	65S-T6	2	0.49	0.01	0.17	nil
		6	0.17	0.03	0.17	nil
		8	0.17	0.02	0.17	nil
	24S-T3	2	2.30	0.33	0.33	0.03
		6	0.49	0.13	0.17	0.01
		8	0.49	0.05	0.13	0.01
B. C. Fir	65S-T6	2	1.81	0.82	0.66	0.02
		6	0.99	0.03	0.49	0.03
		8	0.66	0.03	0.33	0.99
	24S-T3	2	2.14	1.97	0.49	0.07
		6	1.32	0.82	0.49	0.05
		8	0.82	0.66	0.49	0.10
Elm	65S-T6	2	0.08	nil	nil	nil
		6	0.07	0.01	0.03	nil
		8	0.08	0.02	0.05	nil
	24S-T3	2	0.82	nil	nil	nil
		6	0.13	0.01	0.03	nil
		8	0.08	nil	0.05	nil

TABLE 5—Corrosion Rates of Bare and Anodized Pins Embedded in Wood and Immersed in Sea Water

Wood	Alloy	Months	MILS PER YEAR			
			BARE		ANODIZED	
			No Dowels	Dowels and Paint	No Dowels	Dowels and Paint
Mahogany	65S-T6	2	0.33	0.08	0.17	nil
		3	0.33	0.17	0.17	nil
		4	0.49	0.13	0.17	nil
	24S-T4	2	0.66	0.17	0.17	nil
		3	1.15	0.17	0.33	nil
		4	0.49	0.08	0.17	nil
B. C. Fir	65S-T6	2	0.49	0.82	0.66	0.99
		3	0.49	0.33	0.49	0.33
		4	0.33	0.33	0.33	0.33
	24S-T4	2	1.65	0.99	0.82	0.49
		3	1.48	0.66	0.66	0.33
		4	1.15	0.49	0.49	0.17
Elm	65S-T6	2	0.17	nil	0.02	nil
		3	0.17	nil	0.05	nil
		4	0.17	nil	0.03	nil
	24S-T4	2	0.49	0.07	0.15	nil
		3	0.66	0.17	0.12	nil
		4	0.49	0.17	0.17	nil

Sea Water Tests at Harbor Island, N. C.

Three sets of blocks, similar to those exposed in the humidity cabinet, were immersed in sea water at Harbor Island, N. C., for periods of two, three and four months. (Longer immersion periods were not possible due to the strong marine borer action.) Another two sets of blocks were placed just above the high tide line at Harbor Island in a spray zone near a pipe discharge for one year. Corrosion was less than that in the humidity cabinet for a comparable period.

The B. C. fir (see Figure 2) was most corrosive followed by mahogany and elm which confirms the order found in the humidity cabinet tests. 65S-T6 performed better than 24S-T4 and the beneficial effect of anodizing was less than that brought about by the use of dowels and a coating of zinc chromate priming paint. The results are shown in Tables 5 and 6.

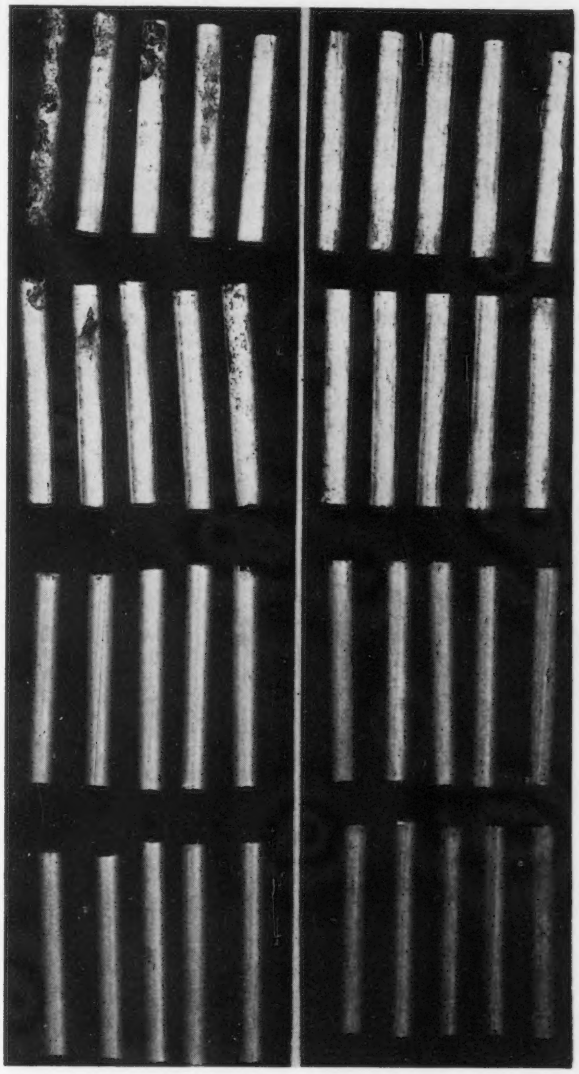

Figure 2—Corrosion of aluminum pins embedded in wooden blocks after two years' exposure to sea water spray at Kure Beach, N. C. (Harbor Island). Wood used was British Columbia fir. Specimens shown at left are Alcan 24S-T4 while those at right are Alcan 65S-T6. In both cases specimens are arranged as follows: top row—bare; second row—bare with zinc chromate and dowels; third row—anodized; bottom row—anodized with zinc chromate and dowels.

The results for the one and two year specimens (Table 6) show that anodizing was very beneficial in all cases and that the use of zinc chromate and dowels was also very helpful but to a lesser extent than anodizing. The performance of both 65S-T6 and 24S-T4 was much better in elm than in the other two woods. This confirms the results of the short term tests.

As would be expected, 65S-T6 was much superior to 24S-T4. It is also noted that corrosion was more severe during the second year.

The stained areas under the open pits on the pins (see Figure 2) showed corrosion penetration which was attributed to intercrystalline attack. The maximum depths of penetration taken at the limits of these areas are given in Table 7.

Long Term Experiments

Five blocks of each wood, except gum wood, were made by fastening two wooden planks 8 x 21 inches together using dowelled 5/8 inch diameter 65S-T5 and galvanized steel bolts (four of each). The dowels were 1 3/8 inches in diameter by 7/8 inch in length with the grain parallel to the surface (see Figures 3 and 4). The aluminum bolts were coated with zinc chromate paint (C.G.S.B.1-GP-40) which was allowed to dry before the bolts were inserted. The galvanized bolts remained unpainted. The dowels were dipped in phenolic resin varnish and forced into the holes while wet. The blocks were wrapped into a rectangular aluminum container in such a way that only the top surface was exposed (see Figure 4), thus simulating actual ship hull conditions. Alumilastic[6] jointing compound of B consistency was used to seal the space between the aluminum box and the wood.

TABLE 6—Average Corrosion Rates* of the Bare and Anodized Pins Embedded in Wood After One and Two Years in Spray Zone at Harbor Island, N. C.

WOOD	Alloy	Condition	MILS PER YEAR			
			Bare		Zinc Chromate and Dowels	
			1 Year	2 Years	1 Year	2 Years
Elm	65S-T6	Bare	0.11	0.20	0.03	0.07
	65S-T6	Anodized	nil	0.02	nil	<0.01
	24S-T4	Bare	0.21	0.58	0.14	0.40
	24S-T4	Anodized	0.03	0.11	nil	<0.01
Elm†	65S-T6	Bare	0.05	.....	0.01	.....
	65S-T6	Anodized	<0.01	.....	nil	.....
	24S-T4	Bare	0.43	.....	0.34	.....
	24S-T4	Anodized	0.02	.....	nil	.....
Mahogany	65S-T6	Bare	0.17	0.05	0.05	0.08
	65S-T6	Anodized	0.01	0.05	nil	0.02
	24S-T4	Bare	0.30	0.94‡	0.18	0.24‡
	24S-T4	Anodized	0.01	0.06	nil	0.02
British Columbia Fir	65S-T6	Bare	0.25	0.35	0.11	0.20
	65S-T6	Anodized	0.13	0.18	0.05	0.12
	24S-T4	Bare	0.77	2.23	0.08	0.90
	24S-T4	Anodized	0.12	0.22	0.23	0.12

* Average of 5 specimens.
† Block which came loose before year was up and was taken from water.
‡ Two year mahogany results average of 10 specimens.

Paint Systems Investigation

The dowelled surfaces of each set of three blocks were given one of the following paint systems:

(a) Two coats of varnish only.

(b) Toxic sealer plus barrier layer plus antifouling paint.

(c) Barrier layer plus antifouling paint.

(d) Toxic sealer plus antifouling paint.

(e) Antifouling paint only.

The three blocks with paint system (a) were then immersed for three months in the Atlantic Ocean at Daytona Beach, Florida, where the water conditions are such that fouling takes place throughout the entire year. A summary of tests is given in Table 8. All other blocks were immersed as above at Daytona Beach. The most corrosive woods remained under exposure for the longest periods. Upon return to the laboratory the blocks were cut open and the bolts cleaned with soap impregnated steel wool (SOS) pads with the exceptions that the three year aluminum bolts were oxide stripped in a chromic-

[6] Parr Paint & Colour Company, Cleveland, Ohio.

TABLE 7—Maximum Depth of Corrosion Penetration in the 24S-T4 and 65S-T5 1/4 Inch Pins After Two Years' Exposure in the Spray Zone at Harbor Island, N. C.

WOOD	Alloy	Maximum Penetration in Mils			
		Bare		Anodized	
		Bare	Zinc Chromate and Dowels	Bare	Zinc Chromate and Dowels
Rock Elm	65S-T6	42	22	24	0
	24S-T4	100	42	45	0
Mahogany No. 1	65S-T6	70	13	18	30
No. 2	65S-T6	43	25	57	28
No. 1	24S-T4	46	184	0	22
No. 2	24S-T4	105	28	0	0
British Columbia Fir	65S-T6	39	40	46	38
	24S-T4	105	130	47	16

TABLE 8—Test Conditions and Performance of 65S-T6 and Galvanized Steel Bolts Exposed in Elm, Mahogany and Fir Blocks

Wood	No. of Blocks	Paint System[1]	Exposure Periods[2]	RESULTS	
				Bolts	Paint
Elm	5	(a)	3 months	Aluminum bolts slightly pitted; galvanized bolts coated with white corrosion product.	Infestation severe; paint system inadequate.
		(b) (c) (d) (e)	10 months	Aluminum bolts in excellent condition; galvanized bolts in good condition.	No infestation with any of the systems; bolts least affected with system (b).
Mahogany	5	(a)	3 months	Both aluminum and galvanized bolts in good condition.	Infestation severe; paint system inadequate.
		(b) (c) (d) (e)	2 years	Aluminum bolts in excellent condition; galvanized bolts rusting increasing with time.	No infestation with any of the systems; bolts least affected with system (b).
Fir	5	(a)	3 months	Head of 1 aluminum bolt pitted—others good; galvanized bolts more heavily coated than in elm.	Infestation severe; paint system inadequate.
		(b) (c) (d) (e)	3 years	Aluminum bolts in excellent condition; galvanized bolts extensive rusting but mechanical properties not affected.	No infestation with any of the systems; bolts least affected with system (b).

Notes: [1] Legend for paint systems:
(a) Two coats of varnish only; (b) toxic sealer + barrier layer + anti-fouling paint;
(c) Barrier layer + anti-fouling paint; (d) toxic sealer + anti-fouling paint; (e) anti-fouling paint only.
[2] The blocks were removed for examination at monthly intervals for the first year, at three month intervals for the second year and after five months of the third year. The examination was visual and by X-ray when necessary.

phosphoric acid solution (ASTM B-137-45) prior to cleaning with steel wool and the three year galvanized bolts were given a five minute dip in 5 percent acetic acid prior to scouring with SOS.

Blocks Exposed 10 Months, 2 and 3 Years at Daytona Beach, Florida

In no case was there serious corrosion of the aluminum bolts. Due to the nature of the test, it was difficult to determine whether the increase in corrosion was due solely to the increased time of exposure or whether the difference in the corrosivity of the woods played a part.

The attack on the galvanized steel bolts was characterized by an increase in rusting with time which effectively masked any difference which might have been expected due to the difference in corrosivity of the woods. The galvanized bolts were badly rusted after three years exposure and practically none of the galvanizing layer was left, although some sections of the iron-zinc intermetallic layer remained. Rusting, although general, was confined to the surface layer and it was felt that the mechanical properties of the bolts had not been adversely affected. The performance of the paint systems with respect to protection of the bolts is shown by consideration of the figures in Table 9.

There was no borer infestation of the blocks through any paint system although some shipworms and boring clams had found means of ingress through the caulked edges of the wooden blocks or where the paint system was not continuous due to mechanical failure.

The complete paint system of sealer + barrier layer + copper antifouling paint, provided maximum protection to the aluminum and galvanized bolts from corrosion. The lack of either or both the sealer or barrier layer produced a significant increase in the corrosion encountered. The lack of the toxic sealer layer produced the least increase in corrosion to the aluminum (40 percent) while the use of antifouling paint directly on the wood increased the corrosion by a factor of 4-5. In general all partial paint systems produced approximately the same increase in corrosion (100 percent) to the galvanized steel bolts. There is some evidence that the use of toxic sealer and barrier layer produced a slight increase in corrosion over that produced by the other two partial paint systems, but it is not enough to indicate a clear cut trend.

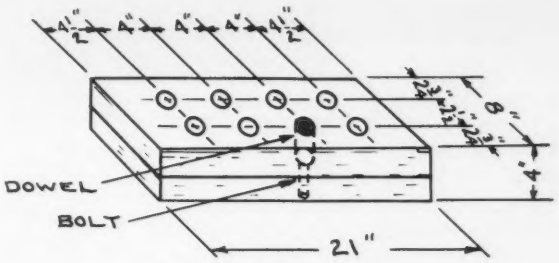

Figure 3—A typical wooden block showing location of the dowels.

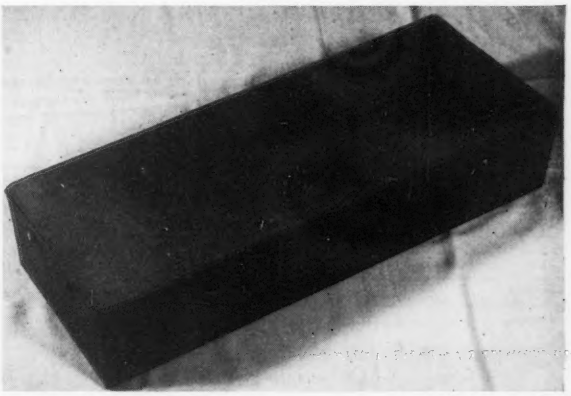

Figure 4—One of the large painted wooden blocks containing four aluminum and four galvanized steel bolts under wooden dowels prior to immersion.

The use of a copper antifouling paint film provided complete protection to all the woods tested from marine infestation for a period of three years while the use of two coats of marine varnish, the original shipyard practice, provides practically no protection to the woods tested from marine borers and shipworms.

Sea Water Pitting Rate Tests

"Strings" of ten coupons each 3 x 6 x 0.125 inch

TABLE 9—Average Percentage of 65S-T6 and Galvanized Steel Bolt Area Corroded (4 British Columbia Fir Blocks Exposed for 3 Years)

Block No.	Paint System	ALUMINUM Head Top	Head Side	Head Under Side	Square Shank	Round Shank	Average Percentage of Total Bolt Area Corroded	GALVANIZED STEEL Head Top	Head Side	Head Under Side	Square Shank	Round Shank	Average Percentage of Total Bolt Area Corroded
5	Toxic Sealer + Barrier Layer + Anti-fouling paint	0.1	nil	0.5	1.8	0.9	3.3	1.1	2.5	3.7	5.2	21.8	34.3
6	Barrier Layer + Anti-fouling paint	2.7	0.3	nil	1.7	0.9	5.6	4.8	7.6	6.6	7.9	27.9	54.8
7	Toxic Sealer + Anti-fouling paint	3.5	0.6	nil	1.6	2.1	7.8	8.7	8.0	6.9	13.8	37.4	74.8
8	Anti-fouling paint	6.5	1.5	nil	0.4	8.1	16.5	1.4	2.5	7.2	10.0	31.0	52.1

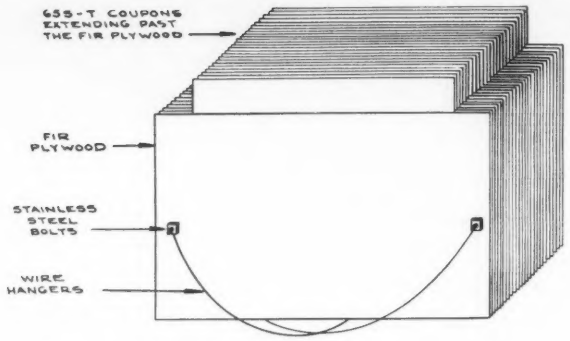

Figure 5—Alcan 65S-T6 aluminum-fir plywood "sandwich."

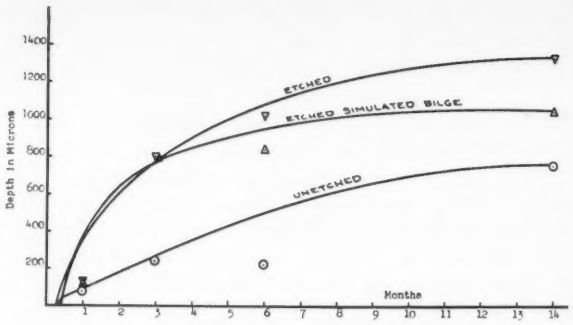

Figure 6—Most probable maximum pit depth on aluminum coupons in fir plywood sandwich assemblies.

TABLE 10—Observations on Sandwich Assemblies of 65S-T6 in Various Seawater Exposures

Condition of Sandwich Assembly	Length of Exposure Period in Halifax Harbor Seawater	Depth of Pits	REMARKS
Unetched	1 week	.....	All coupons contained small blisters but no measurable pits.
Unetched	1 month	3 mils.	Pits on only three coupons. One half surface of coupons showed etched appearance; remainder retained original surface appearance.
Unetched	3 months	Max. 10 mils.	Pits on only a few coupons. Outside surface of No. 1 coupon showed larger number pits (plywood bulged in this sandwich).
Unetched	6 months	Max. 9 mils.	Only very few pits; coupons in very good condition.
Unetched	14 months	Max. 31 mils.	Outside coupon (No. 10) severely pitted (1 pit 48 mils. deep); all others in very good condition.
Etched	1 week	.....	Exposed areas free from pits. Area covered with wood on coupons 1 and 2 showed some staining.
Etched	1 month	Max. 5.5 mils.	All coupons showed large number of very small blisters and shallow pits. (100 pits uniformly over each side).
Etched	3 months	Max. 32 mils.	Only few pits except along edges protruding from plywood (along metal—plywood joining line).
Etched	6 months	Max. 40.5 mils.	Pits evenly over entire surface; more small, shallow pits on exposed surfaces.
Etched	14 months	Max. 53 mils.	Many deep pits of large cross-section along edge of plywood; more numerous pits on boldly exposed surfaces.
Assembly in Simulated Bilge	1 week	Not measurable	Similar to etched coupons.
Assembly in Simulated Bilge	1 month	Max. 4.5 mils.	Similar to etched coupons except blistering not prevalent.
Assembly in Simulated Bilge	3 months	Max. 32 mils.	Similar to etched coupons. Large number pits on areas protruding from plywood; areas covered by plywood showed few pits. Coupons Nos. 1 and 10 more pits on outside surfaces.
Assembly in Simulated Bilge	6 months	Max. 33 mils.	Similar to those immersed for 3 months. Shallow pits for most part but each coupon showed a few deep pits.
Assembly in Simulated Bilge	14 months	Max. 42 mils.	Better condition than etched totally immersed. Some uniform etch type corrosion between coupons and plywood. Very small pits over entire area; greater concentration along edge of wood. Outside area coupons 1 and 10 showed more larger pits.

of Alcan 65S-T6 sheet with each coupon sandwiched between strips of ⅛ inch thick fir plywood with a portion of each coupon protruding from one side, were prepared. The strips of fir plywood protruded beyond the ends of the coupons. Stainless steel bolts were used to hold the coupons tightly in place between the strips of plywood (see Figure 5). Wire holders were attached to the bolts so that the sandwiched strings of coupons could be suspended easily in sea water. The strings were immersed in sea water in Halifax Harbor, N. S. (Atlantic Ocean), for periods of one week, one, three, six and fourteen months, under the following test conditions:

1. One string for each time period was immersed in the sea water with the surface of the metal in its existing condition (that is, not etched).
2. One string for each time period was etched in 85 percent phosphoric acid at 70 C for two minutes and rinsed in flowing sea water; this was done at the location where they were immersed. They were immersed for testing within one hour from the time they were etched. Each etched string was dismantled prior to etching and reassembled after etching.
3. An additional set of strings was etched and placed in a shallow box, partially filled with sea water, in a barge in the harbor so that wetting and drying action of the sea water on the sandwiched strings would occur due to the movement of the barge. The sea water was changed weekly. This was considered to be a "simulated bilge" condition.

After each time period the strings were removed and dismantled and the coupons numbered consecutively as they were arranged in the sandwiches. After drying they were returned to the laboratory in Kingston, for evaluation.

The coupons on all the strings were cleaned in chromic-phosphoric acid stripping solutions (ASTM B-137-45) and examined under a low powered microscope (7X) for pitting and corrosion. If pits were present on the coupons they were measured for depth by a calibrated higher powered microscope (125X).

Figure 6 shows the pitting rate curves for the etched and unetched sets of sandwich strings exposed in Halifax Harbor sea water and the etched sandwich strings exposed in simulated bilge conditions. The points through which the curves are drawn were derived from the calculated maximum pit depth obtained from extreme value plot analyses.[1]

Further results are shown in Table 10.

Pits in Alcan 65S-T6 were more numerous in the case of the etched "sandwich" string, while pits were fewer and more shallow on the unetched string. Pitting was greater along the line where the aluminum panels protruded beyond the plywood. This probably was due to a differential aeration caused by the unexposed area at the edge becoming more cathodic than the boldly exposed protruding area.

The simulated bilge conditions did not cause greater corrosion of the aluminum; in fact, the reverse was true, since pitting was somewhat less in depth than in a similarly prepared string totally immersed in the sea water.

Summary of Results

The results of these experiments indicate that:

1. The relative corrosivity to aluminum of the woods tested under ship hull conditions is: rock elm (least), mahogany and B. C. fir (most). Only very slight corrosion was encountered, however, on chromate primed bolts, even in the most corrosive wood after three years immersion in sea water. This suggests that chromate primed and dowel covered 65S-T6 aluminum hull bolts will have a long and adequate life ($>$ 10 years).
2. As expected 65S-T6 has a much superior corrosion resistance to a marine environment than 24S-T3 & 4. This is much more apparent in the long term results. Short term results show 65S-T6 to be twice as resistant as 24S-T3 & 4 while the average long term increase in resistance is greater than four times that of 24S-T3 & 4 as shown by two year results.
3. Anodizing increases the corrosion resistance of both alloys, particularly if this treatment is followed by a coat of zinc chromate primer. This increase is again more striking in the long term results which show anodized specimens to have as much as 2-5 times greater corrosion resistance than the comparable bare samples. This is in contradistinction to the earlier short term results in which an improvement showed which appeared to lessen with time.
4. The use of wooden dowels to protect aluminum embedded in these woods is beneficial and in general increases the resistance to corrosion by a factor of two. This is a significant amount and it is strongly recommended that wherever aluminum bolts are to be embedded in wood immersed in sea water the bolts first should be given a coat of zinc chromate primer and after embedment they should be capped with wooden dowels.

   A further example of the improvement in corrosion resistance gained by using this system was apparent from the Florida exposure of the large wooden blocks. A comparison of the corrosion encountered on the unpainted interior of the containers with that of the aluminum bolts within the blocks, which had been protected with a coat of zinc chromate primer and wooden dowels again shows the advantage gained by these additions.
5. Where pitting does occur on 65S-T6 embedded in sea water wetted wood it has been shown that the rate of penetration falls off sharply with time. This behavior is similar to the pitting of aluminum alloys in fresh water,[2] although the rate is higher (about double for 65S-T6—sea water vs Kingston Tapwater).[3]

References

1. P. M. Aziz. Application of the Statistical Theory of Extended Values to the Analysis of Maximum Pit Depth for Aluminum. *Corrosion*, **12**, 35-46 (1956).
2. T. E. Wright and H. P. Godard. Laboratory Studies on the Pitting of Aluminium in Aggressive Waters. *Corrosion*, **10**, 195-198 (1954).
3. Aluminium Laboratories Limited, Kingston. Unpublished work.

Any discussions of this article not published above will appear in the December, 1957 issue.

PREVENTIVE MEASURES, CA

Partial protection is also very desirable in many cases involving poorly coated lines and can be justified by reductions in sinking fund deposit factors due to increased pipe life, in addition to usual savings in operations.

Electrical engineers should anticipate that cathodic protection will effect savings in other corrosion areas. Power plant equipment, cables, substations, docks, ships and other situations involving metals in contact with soils and solutions are fertile fields for the application of cathodic protection and modern techniques of corrosion control

5.2.1-56 CORROSION CONTROL OF STEEL EQUIPMENT. T. R. B. Watson. **Can. Chem. Processing, 37,** No. 10, 46, 48, 50, 52, 54 (1953) Sept.

Describes causes of corrosion and discusses control by means of cathodic protection.

5.2.1-57 CATHODIC PROTECTION LICKS CORROSION. L. C. Werkin. **Petroleum Refiner, 31,** No. 9, 122-124 (1952) Oct.

Cathodic protection, a positive, controllable and inexpensive method of preventing corrosion. There is real money in reducing these losses caused by corrosion-maintenance, replacement costs, over-design of equipment, and product losses. Refinery management and technical personnel must be keenly aware of the creeping inroads of corrosion, and alert to any feasible solution to the problem. Table showing cost estimates and savings resulting from its use on a large open-box-type refinery condenser and a large diameter Dorr clarifier. 9 refs.

...THODIC PROTECTION OF STEEL. F. ...Nurse, H. C. K. Ison and T. W. Farrer. ...ry, No. 40, 972-974 (1952) Oct. 4.

...rrent and potential requirements for ...teel in sea water and in the presence ... Protection of mild steel in sea ...tly stages at a relatively noble ...nsity is sufficiently high, ...gh a steel anode. If a ... protection can be ... small current ...esence of the

1.6–3, 4; **4.5.1–1; 4.5.3-6.3.8–6; 7.2–17, 44; 8.4.2–8; 8.4.3-8.9.5–12**

5.2.2 PREVENTIVE PROTECTION, ANC

a. General
b. Magnesium

5.2.2-1 CATHODIC PROT ANODES. Brochure, June, 1953 tute, 60 East 42nd Street, New

Merits of zinc anodes (con and with rectified systems) in ing distribution piping, tra condensers. Practical

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Technical Practices Committee Has Charge of Policy

The Technical Practices Committee of the National Association of Corrosion Engineers is composed of a chairman, vice-chairman and the chairmen of all Technical Group Commitees. The vice-chairmen of group committees are alternate members. The president, vice-president and executive secretary of NACE are members ex-officio of the Technical Practices Committee.

The chairman and vice-chairman are appointed by the president of NACE for two year terms.

The Technical Practices Committee coordinates functioning of all technical committees. It establishes procedures necessary for orderly business conduct of the committees. It endeavors to stimulate committee activity and aids in initiation of new activities. Through its chairman it makes recommendations to the board of directors for changes or powers to more effectively achieve the aims of technical committees. In general, it advises and acts on matters of policy and planning affecting the technical committees.

Members of the Technical Practices Committee are E. C. Greco, United Gas Corporation, Shreveport, Louisiana, chairman; T. J. Maitland, American Telephone & Telegraph Co., New York, New York, vice-chairman, and the following group committee chairmen: Jack L. Battle, Humble Oil & Refining Co., Houston, T-1; W. H. Stewart, Sun Pipe Line Company, Beaumont, Texas, T-2; A. H. Roebuck, Continental Oil Company, Ponca City, Oklahoma, T-3; Irwin C. Dietze, Dept. of Water & Power, Los Angeles, California, T-4; R. I. Zimmerer, Petro-Tex, Inc., Houston, Texas, T-5; L. S. Van Delinder, Union Carbide Chemicals Co., South Charleston, West Virginia, T-6.

Vandelinder

Zimmerer

Members ex-officio of the Technical Practices Committee are: W. H. Stewart, president; L. L. Whiteneck, vice-president; A. B. Campbell, executive secretary.

Alternate members of the Technical Practices Committee are: W. C. Koger, Cities Service Oil Company, Bartlesville, Oklahoma, T-1; L. P. Sudrabin, Electro Rust Proofing Corp., Newark, New Jersey, T-2; E. P. Teale, American Telephone & Telegraph Co., Washington, D. C., T-4.

Elections currently are being held to elect vice-chairmen for group committees T-3, 5 and 6.

The managing committee of the Tech-

(Continued on Page 88)

Informal Report Is Made by T-4D on Tests Being Made

It is estimated that over two million tons of salt for deicing will be sold to cities in the United States in 1957. This figure was cited to Unit Committee T-4D on Corrosion by Deicing Salts at the committee's meeting held in St. Louis. To emphasize the increase in salt sales, figures for 1949 were given as 400,000 tons; 1953 sales were 800,000 tons and in 1955 1,500,000 tons of salt were sold.

The T-4D task group for coordinating field programs between cities and transportation companies did not make a formal report to T-4D at the St. Louis meeting. However, an informal summation of data obtained by the task group during the winter of 1955 and 1956 was given. During 1955 and 1956 field corrosion tests were carried out by several municipalities in cooperation with a manufacturer of inhibitors.

City Vehicles Are Used

Test coupons were mounted on police cars and other city vehicles using both painted and unpainted 3 x 3-inch cold rolled steel panels. In most cases the panels were bolted to gravel deflector plates underneath the front fenders of vehicles. In a few instances the panels were suspended below and just behind front bumpers. From four to eight test panels were installed on each test vehicle. The number of vehicles varied from a low of three at one city to a maximum of twenty-two.

Corrosion rates were determined from weight loss data for unpainted panels only for exposure periods varying from 39 to 103 days. There were so many uncontrollable variables involved in field tests of this nature it is difficult to arrive at any firm conclusions in analyzing weight loss data for coupons exposed in areas where salt with a corrosion inhibitor was used as compared to areas where uninhibited salt is used. However, it was reported that despite these recognized limitations the data show a very definite trend in favor of inhibited salt.

Inhibitor Ratio Varies

Normally, it is recommended that a ratio of one pound of inhibitor should be used for each 100 pounds of salt. Definite benefit can be expected at a ratio as low as 0.5 inhibitor while optimum results can be expected at a 2 percent inhibitor to salt ratio. As an illustration, one city using only 0.44 percent inhibitor showed an average total rate loss per 1000 miles of vehicle travel that was 29 percent lower than in an adjacent community using untreated salt. If it is assumed that roughly 50 percent of the total weight loss is caused by corrosion effects of the salt only it would follow that the salt-induced corrosion was 58 percent lower in the community with

(Continued on Page 86)

Committees Allocated Full Day at Pittsburgh

A full day of technical committee meetings has been scheduled for the North East Region meeting to be held in Pittsburgh, Pennsylvania at the Penn-Sheraton Hotel November 12-14. Technical committee meetings will be held all day on November 12, beginning at 9 a.m. The tentative schedule of technical committee meetings is:

T-5B on Plastic Materials of Construction.

T-6F on Protective Interior Linings Applications and Methods.

T-2E on Internal Corrosion of Product Pipelines and Tanks.

T-3C on Annual Losses due to Corosion.

T-2K on Prefabricated Plastic Film for Pipeline Coating.

T-6D on Industrial Maintenance Painting.

T-7A Northeast Region Corrosion Coordinating Committee.

T-5A-6 on HF Corrosion.

T-3E-1 on Corrosion of Railroad Tank Cars.

T-3H Tanker Corrosion.

Over 2,000,000 Tons—

(Continued From Page 85)

inhibited salt even though used at less than half the recommended concentration.

It was reported that the cost of using an inhibitor at a dosage of one percent is roughly 20 to 25 cents per motor vehicle for a given community. Transportation companies were not used for field testing purposes during the 1956-1957 winter season and no definite plan had been made yet the for the 1957-1958 season.

The committee discussed the fact that on wrapped cables under cathodic conditions, corrosion continues from cathodic products at the sheath after current has ceased to flow. One member voiced an objection to use of the term "cathodic corrosion" and suggested that instead the term corrosion by "cathodic product" be used.

Manhole Tests Reported

The committee also discussed possible effect on underground equipment by increased use of deicing salts. A report was given by a member of the committee on manhole water chlorine tests made in April 1956 as compared with tests made in February and March of 1957. Samples taken from one manhole on April 26, 1956, revealed chlorine content to be 4092 ppm and chlorine as NACL gms/gal. to be 210.23.

One company reported that the number of cable failures in the city where his company operates is increasing and that salt is a factor. He has recommended that all cables beyond a certain size be covered with a protective jacket.

The question was asked the committee if salt without potentials would cause corrosion. A discussion indicated that alkalinity at the surface of a cable causes corrosion without stray current.

One member said lead cables passing through areas in the earth where there are heavy salt concentrations and where there are light or no concentration would be effected by long cell action causing anodic corrosion.

Another member said that in his city manholes are sealed to keep surface water out. He also said that a survey had been made in some cities where manholes were not sealed. He said he was of the opinion that it was unnecessary for his company to continue sealing manholes but after hearing discussions at the T-4D meeting he decided to continue to seal them.

Polyurethane Group Is Headed by Sansone

Richard S. Sansone, Mobay Chemical Company, St. Louis, Mo. has been appointed chairman of Task Group T-6A-17 on Polyurethanes. Mr. Sansone is a development specialist for the Mobay Chemical Company. He received a BS degree in chemistry from McMaster University in Hamilton, Ontario in 1951. Mr. Sansone concentrates on the development of markets for urethane coatings, working directly with paint manufacturers and surface coatings formulators in the development of commercial urethane coating systems for specific industrial applications.

Sansone

Before joining Mobay he was associated with Canadian Industries Ltd. where he held the positions of works chemist, technical service representative and market development specialist in organic chemicals.

Any NACE member interested in actively working on the polyurethane task group is invited to write Mr. Sansone at the Mobay Chemical Company, 1700 South 2nd St., St. Louis 4, Mo.

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Twenty-Six Technical Committees to Meet At Oklahoma City During Regional Session

Twenty-six technical committees have scheduled sessions during the South Central Region meeting in Oklahoma City, October 1-4. The tentative schedule is as follows:

T-1, Corrosion in Oil and Gas Well Corrosion.
T-1B, Condensate Well Corrosion.
T-1D, Sour Oil Well Corrosion.
T-1F, Metallurgy.
T-1F-1, Sulfide Stress Corrosion Cracking.
T-1F-2, Sweet Crude & Sweet Condensate Stress Corrosion Cracking.
T-1H, Oil String Casing Corrosion.
T-1K, Inhibitors for Oil and Gas Wells.
T-1M, Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations.
T-2C, Minimum Current Requirements.
T-2D, Standardization of Procedures for Measuring Pipe Coating Leakage Conductance.
T-2F, Internal Corrossion of Crude Oil Pipe Lines and Tanks.
T-2G, Coal Tar Coatings for Underground Use.
T-2J, Wrappers for Underground Pipe Line Coatings.
T-2K, Prefabricated Plastic Film for Pipe Line Coating.
T-3D-1, Electrical Holiday Inspection of Coatings.
T-3G, Cathodic Protection.
T-5C-1, Corrosion by Cooling Water.
T-5E, Stress Corrosion Cracking of Austenitic Stainless Steels.
T-6A, Organic Coatings and Linings for Resistance to Atmospheric Corrosion.
T-6B, Protective Coatings for Resistance to Atmospheric Corrosion.
T-6D, Industrial Maintenance Painting.
T-6E, Protective Coatings in Petroleum Production.
T-7D, South Central Region Corrosion Coordinating Committee.
T-7D-1, Houston Section Corrosion Coordinating Committee.
T-8, Refining Industry Corrosion.

Los Angles Oil Equipment Committee Elects Kipps

H. J. Kipps, Signal Oil and Gas Company, Long Beach, Cal. and L. W. Brock, Long Beach Harbor Department, Long Beach have been elected chairman and vice-chairman respectively of NACE Technical Unit Committee T-1A on Corrosion in Oil and Gas Well Equipment, Los Angeles Area.

Unit Committee T-1A holds monthly meetings to discuss various aspects of oil and gas well equipment. The meetings vary from formal presentation of papers to informal round table discussions. The scope of the committee includes that of all other unit committees under Group Committee T-1. Work of the Los Angeles area committee and other T-1 committees is coordinated through liaison representatives.

Thomas Is Elected by Domestic Water Group

J. F. J. Thomas of the Industrial Waters Section of the Department of Mines and Technical Surveys in Ottowa, Ontario, Canada has been elected vice-chairman of Unit Committee T-4E on Corrosion by Domestic Waters.

T-4E has been testing the corrosivity of various domestic waters for the past two years. It is expected a report on the tests will be available next year. T. E. Larson, Illinois state Water Survey is chairman of the committee.

Munger

Pittman

Draft of Report on Coal Tar Base Linings Completed

Task Group T-6A-16 on Bituminous Coatings for Resistance to Chemical Corrosion has completed the first draft of a report entitled "Coal Tar Base Linings." Dr. C. U. Pittman, Koppers Company, Inc., Tar Products Division, Verona, Pa., chairman of T-6A-16, recently submitted the draft to C. G. Munger, Amercoat Corporation, chairman of Unit Committee T-6A. Mr. Munger has submitted the report to membership of T-6A for review and comment. The committee will hear a verbal presentation of the report at the meeting to be held at Oklahoma City during the South Central Region meeting October 1-4.

The report defines coal tar base linings as a class of materials in which protection of the substrate from corrosion is provided largely by coal tar pitch. Coal tar base linings are available for hot or cold application. For the former the material is melted before use and applied in the molten form; it returns to its original condition when cooled. When the lining is to be applied without heating it is made to the required consistency by the incorporation of a coal tar distillate having the desired boiling range.

The report describes uses for coal tar linings and presents a summary of physical properties.

Hot applied base linings are reported to be available in a range of physical properties but the so-called "wide range enamel" is most generally used. They can be applied to the inside of the pipe by spinning to give a uniform, smooth, glossy lining and to other metal surfaces by hand daubing. The report also reviews the effect of compounding and lists resistances of the linings. A section of the report covers surface preparation, priming, application methods, and coverage.

Technical Practices—

(Continued From Page 85)

nical Practices Committee is the executive group of the NACE technical committee organization. It conducts business for the Technical Practices Committee between meetings of the latter. Matters of policy and recommendations for guidance of technical committee affairs usually are considered first by the managing committee and then referred with recommendation to the Technical Practices Committee.

The managing committee is composed of the chairman and the vice-chairman of the Technical Practices Committee and the vice-president of NACE.

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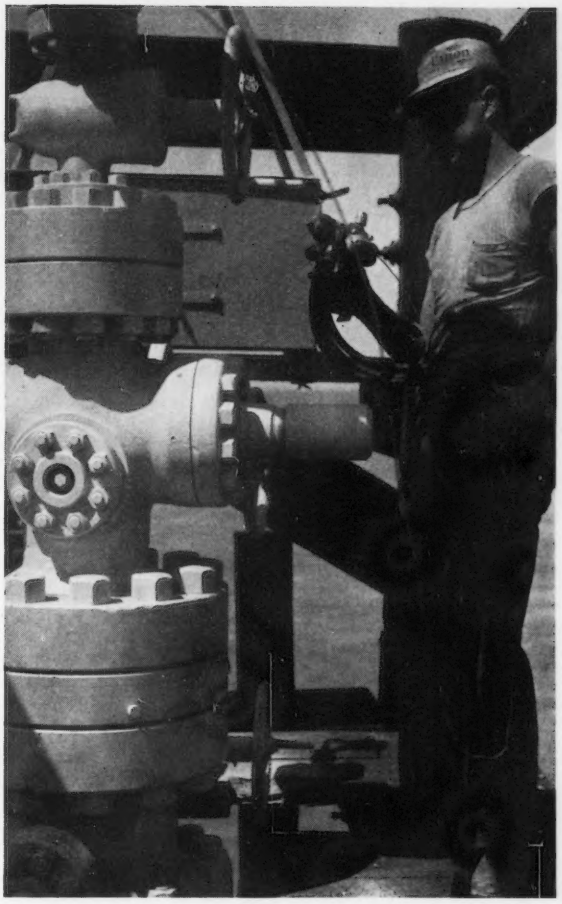

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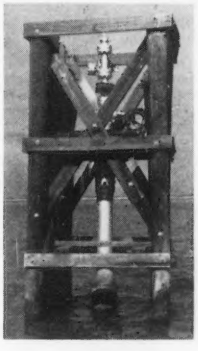

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Maximum Tank Car Corrosion Rate Discussed

Maximum permissible corrosion rate for a steel railroad tank car is 0.05-0.0 ipy. This was the consensus at a recen meeting of Unit Committee T-3E o Railroads. When this rate is exceeded a new lining, cladding or replacement o the tank with one of a different alloy i indicated.

J. R. Spraul, General American Trans portation Corp., chairman of T-3E-1 o Railroad Tank Cars reported to T-3E on field data compiled on tank car lining failures and presented a list of definitions of what constitutes failure. The group had noted previously that tank cars i service with one commodity may require extensive preparation before a lining re sistant to another commodity can b applied.

The task group is considering preparation of a chart giving instructions and suggestions for cleaning lined tank cars.

The committee noted that most corrosion occurs in empty cars when vapor from residual product, mixed with water vapor and relatively high temperatures combine to attack interior steel walls.

Task Group T-3E-2 on Railroad Hopper Cars has reported on stationary hopper car tests in which strip magnesium was placed around the interior of a small storage hopper carrying a relatively high sulphur coal. Tests show that hoppers combining copper-bearing steel and low alloy steel especially designed for this service were perforated after 170 days' exposure. Similarly constructed hopper cars lined with magnesium and exposed for 598 days continue to show excellent integrity. It is estimated the cars probably could continue in good condition for another 300 days provided the magnesium is consumed at a steady rate. Details of the test will be published soon.

More Data Sought on High Pressure Oil Wells

Task group T-1B-1 on High Pressure Wells has held two meetings since the annual conference in St. Louis. A questionnaire on typical high pressure well completion was prepared at the first meeting of the task group. The results of the questionnaire will be reported at the T-1B Condensate Well Corrosion Committee meeting to be held in Oklahoma City next October.

W. F. Oxford Jr., Sun Oil Company Beaumont, Texas, invites all those interested in high pressure well corrosion and corrosion mitigation procedures to send available data to him prior to the Oklahoma meeting.

Industrial Research Meeting Held at Columbia U

The Eighth Annual Conference on Industrial research sponsored by the Department of Industrial and Management Engineering of Columbia University was held June 3-6 at Harriman, N. Y. Data discussed included research expectations, communication, information handling and computers.

•

NACE aims to use CORROSION as a medium for coordinating corrosion control work the world over.

NACE NEWS

35 Papers Listed for North Central Region Meet

NEW CHICAGO SECTION OFFICERS, left to right: I. J. Acosta, Crane Company, secretary; V. M. Kalhauge, Standard Oil Co. of Indiana, chairman and D. G. Keefe, Swift & Co., treasurer. H. C. Boone, vice-chairman, was absent when the photograph was taken.

October 1-4 Sessions At Chicago Include An Exhibition

Titles, some tentative, for 35 papers to be presented during the October 1-4 North Central Region meeting at Chicago have been released. The meeting, with concurrent exhibition, will be held at the Sherman Hotel. More papers will be accepted before the program is complete.

In addition to the symposia the schedule calls for technical committee meetings all day October 1.

The tentative schedule has been given as follows:

Tuesday, October 1

Technical committee meetings all day.

Wednesday Morning, October 2

Protective Coatings Symposium, C. A. VanNatta, Plastic Linings Coatings, chairman.

Underground Corrosion and Its Mitigation by the Use of Coal Tar Coatings, F. J. Kiernan, Barrett Division, Allied Chemical & Dye Corp.

Protective Coatings in Use With Cathodic Protection, F. Ploederl, Wisconsin Protective Coatings Co.

Classifying Protective Coatings and How to Evaluate the Base (Resin) Formulations for Maintenance Purposes, L. Gleekman, Wyandotte Chemical Co.

Water Treating Symposium, R. G. Rydell, National Aluminate Corp., chairman.

Practical Solution of Cooling Water Problems, T. Harding, Omaha Public Power District.

Aluminum Cooling Towers and Their Treatment, A. J. Haygood and J. Midford.

Results of a Study on How Mineral Content of Water Affects Corrosion, T. E. Larson, Illinois State Water Survey.

Correlation of Laboratory with Field Work on Cooling Waters, H. M. Steininger, Standard Oil Co. of Indiana.

Wednesday Afternoon, October 2

Protective Coatings Symposium—continued.

Chemically Preparing Metal Surfaces for Coatings, by Stochbower, American Chemical Paint Co.

Commercial Spray Equipment, Including Hot Spray, Heavy Duty Materials, Low Pressure and High Pressure Spraying and Equipment Maintenance, W. R. Brooks, Binks Mfg. Co.

Discussion and question period on subjects covered.

Pipe Line Corrosion Symposium, J. L. Adkins, Peoples Gas Light and Coke Co.

Inhibitor Testing for Products Pipe Lines, R. H. Meyer, Sinclair Pipeline Co.

(Continued on Page 92)

Permian Basin Tour Scheduled October 23-25

The Seventh Biennial Permian Basin Corrosion Tour, sponsored by Permian Basin Section NACE will be held October 23-25 in the Midland-Odessa area. The tour features inspections of materials, tanks and other equipment and discussions on the relative effectiveness of preventive measures.

At the May 1 meeting of Permian Basin Section held at Ranchland Hills Country Club, Midland, 68 members and guests heard C. C. Nathan of the Texas Company speak on corrosion inhibitors.

Next meeting of the section was scheduled to be held June 5 at the Lincoln Hotel in Odessa.

Wachter, Smith Join Policy And Planning Committee

Carl B. Smith, Dearborn Chemical Co., Chicago and Aaron Wachter, Shell Development Co., Emeryville, Cal. have accepted posts on the NACE Policy and Planning Committee. They replace Vance N. Jenkins, American Petroleum Institute, New York and J. J. Pokorny, Cleveland Electric Illuminating Co. whose terms expired in March.

E. P. Noppel, Ebasco Services, Inc., New York, chairman said an effort is made to make the committee representative of the association on both geographical and industrial bases.

Other members of the committee are: L. B. Donovan, Consolidated Edison Co. of New York; John E. Loeffler, Thornhill-Craver Co., Inc., Houston; R. A. Davis, Chicago Bridge & Iron Co., Birmingham, Ala. and H. P. Godard, Aluminium Laboratories, Ltd., Kingston, Ont.

EXHIBIT SPACE ORDERS BEING RECEIVED NOW

Orders for exhibit space at Chicago's Sherman Hotel and Oklahoma City's Municipal Auditorium are coming in rapidly at Central Office NACE. The exhibitions are to be held during the meetings of North Central Region at Chicago and South Central Region at Oklahoma City, both October 1-4.

Contracts and exhibit specifications, including floor plans were mailed from Central Office late in May to lists of prospects for both exhibitions. Prospective exhibitors interested in showing at either or both events may get copies of the material from Central Office on request. Exhibit committees of the two regions are working to make both events sellouts.

Although South Central Region held a successful exhibition in connection with its 1956 meeting at San Antonio, this year's show at the Sherman Hotel is the first held by North Central Region. The 1956 exhibition at San Antonio was the first held by NACE other than at the national level.

Regional exhibitions were organized in an effort to answer the requests from companies expressing an interest in regional displays only, often in preference to the annual national exhibition. The association seeks to give manufacturers and others opportunities to show their products to groups with geographical orientation related to the companies' sales areas.

Shreveport Section

Shreveport Section has scheduled 12 meetings in 1957, all on first Tuesdays monthly at the Washington-Youree Hotel, beginning at 6 pm.

Hugh L. Hamilton Resigns

Hugh L. Hamilton, A. V. Smith Engineering Co., Narberth, Pa. has resigned from the National Association of Corrosion Engineers' board of directors as a member representing active membership. The board of directors will select a replacement.

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Panel on Petroleum Fuel Corrosion Held at New York

Fifty-one members and guests attended the Metropolitan New York Section meeting May 7 at the Chi-Am Chateau, Mountainside, New Jersey. R. W. Manuel, Socony Mobil Oil Company, New York City acted as moderator for a panel discussion on "Corrosion Problems Arising from the High Octane Race, Upgrading Fuel Oil and Treating Foreign Crudes." The panel was composed of the following: G. Sorrell, M. W. Kellogg Company; R. D. Merrick, Esso Research & Engineering Company and S. J. Artese, Shell Oil Company. Following are highlights of the discussion: High temperature hydrogen sulfide corrosion, high temperature hydrogen attack, acid gas scrubber corrosion, acid chloride corrosion in hydrogenation systems and injection of inhibitors and neutralizers in refinery units.

This was the last meeting for the current season. Next meeting is scheduled for September in the New Jersey area.

35 Papers Listed–

(Continued From Pame 91)

Corrosion Protection for Pipe Type Transmission Lines, W. T. Rose, Commonwealth Edison Co.

Asphalt Coating for Pipe Line Protection, D. C. Palm, Natural Gas Pipeline Co. of America.

Tape Coatings in Pipeline Construction, H. D. Segool, Polyken Products, The Kendall Co.

Thursday Morning, October 3

Cathodic Protection Symposium, G. G. Wilson, Institute of Gas Technology, chairman.

Experimental Studies of Cathodic Protection of Naval Vessels, L. J. Waldron, M. H. Peterson and M. C. Bloom, U. S. Naval Research Laboratory.

Measurement of Cathodic Protection, L. P. Sudrabin, Electro Rust-Proofing Corp.

Evaluation of Protective Coatings Under High Potential, D. F. Koenecke and R. I. McDougall, Esso Research and Engineering Co.

Demonstration of the Theory of Cathodic Protection, W. J. Kretschmer, Columbia Gas System Corp.

Transportation Symposium, S. K. Coburn, Association of American Railroads, chairman.

Corrosion in Tankers, F. M. Watkins, Sinclair Petroleum Laboratories.

Laboratory Testing of Railroad Diesel Cooling System Corrosion Inhibitors, J. I. Bregman and D. B. Bores.

Some Aircraft Corrosion Problems, H. L. Stevens, Wright Air Development Center.

Conductivity Testing of Zinc Dust Pigmented Coatings—Test Methods for Evaluating Conductivity of Zinc Rich Paints, Lt. D. S. Nantz, Wright Air Development Center.

Thursday Afternoon, October 3

Cathodic protection round table discussion.

Chemical Processing Industries Symposium, W. A. Watkins, The Duriron Co., Inc., chairman.

Corrosion in the Corn Wet Milling Industry, R. E. Pierson, F. J. Holsinger and R. Benes, Corn Products Refining Co.

Liquid Metal Corrosion Experience, F. A. Smith, Argonne National Laboratory.

Accelerated Condensation Corrosion Test for Evaluating Rust Preventives, B. A. Dieman and J. W. Gaynor, Standard Oil Co. of Indiana.

Friday Morning, October 4

Refinery Corrosion Symposium, A. Dravnieks, Standard Oil Company of Indiana, chairman.

High Temperature H_2S Corrosion of Stainless Steel, E. B. Backensto, J. E. Prior and J. W. Sjoberg, Socony Mobil Oil Co., Inc.

Corrosion During Chemical Cleaning in Refinery Equipment, K. R. Walston and A. Dravnieks, Standard Oil Co. of Indiana.

Corrosion in Phenol Extraction Units, G. A. Marsh and B. Schaschl, Pure Oil Co.

Public Utilities Symposium, H. B. Kroon, Illinois Bell Telephone Co., chairman.

Cathodic Protection of Electrical Generating Stations, A. W. Peabody, Ebasco Services Inc.

Saturable Reactors for Control Rectifiers, H. E. Kroon, Illinois Bell Telephone Co.

Soil Box Demonstration—M. C. Miller, Ebasco Services Inc.

Use of Plastic Pipe in the Gas Distribution System of Chicago, T. R. Bogumill, Peoples Gas Light and Coke Co.

Friday Afternoon, October 4

Educational Lectures, H. T. Francis, Armour Research Foundation, chairman.

Electrochemistry of Corrosion—Some New Ideas, M. Stern, Electro Metallurgical Co.

Some Unusual Effects of Hydrogen in Corrosion Reactions, J. E. Draley, Argonne National Laboratory.

Electrochemical Mapping of Corroding Surfaces, H. T. Francis, Armour Research Foundation.

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Deep Well Pump Corrosion Is Topic At San Diego Session

A well-received experiment in conducting a section meeting was presented at San Diego May 15. In addition to the principal technical discussion, ten-minute talks by representatives of firms describing new anti-corrosion products and techniques developed by their companies were presented.

The short talk feature may be repeated at future meetings to keep members informed regarding products and techniques which may be valuable to them in their work, according to Kenneth R. Christy, section secretary-treasurer.

The principal technical subject of the meeting was pump corrosion in deep well applications. Corrosion difficulties encountered in deep well pumps used in the Tiajuana River Valley and at National City were described by Norman Beenfeldt, California Water and Telephone Co., Chula Vista, Cal. In addition to Mr. Beenfeldt, Linn Magoffin and Henry Myers, both of California Water and Telephone Co. also participated in the discussion. Mr. Magoffin is chairman of NACE Western Region.

Galvanic Action in Pumps

Types of pump corrosion found included galvanic action due to dissimilar metals, entrapped oxygen and abrasion from sand. One well had corrosion from negative non-carbonate hardness. Protection methods including magnesium anodes and paint coatings have been tried with varying results.

Mr. Myers, the company's chief chemist and sanitary engineer, discussed water conditions along the Pacific Coast and the mineral content affecting or resulting in corrosion. Mr. Magoffin, who instituted some of the protective measures used, said in his opinion, a carefully applied coating gave the best results. He felt that pumps and casings should be pulled frequently for inspection and repair.

Speakers for the brief ten-minute talks were: Bob Hawkins, The Alsynite Co.; Ben Eggert, Frost Engineering Service; Al Stetson, Solar Aircraft Co. and George Taitt, G. Taitt & Co. The meeting was held at Lawton's Restaurant, La Mesa, Calif.

Several Products Discussed

Mr. Hawkins spoke in fiberglass products. Normal mixture is 40 percent fiberglass to 60 percent polyester resins, which produces a light but hard product. However, specifications can be varied to produce desired properties. They have good acid resistance, medium alkali resistance but poor solvent resistance, according to Mr. Hawkins. Principal products of his firm are sheet and corrugated products.

Mr. Eggert described new tape products which are combinations of butyl rubber with polyvinyl chlorides. These tapes are non-porous, bond well to steel, have good aging characteristics and are free from holidays when applied. They are recommended for use on pipe after a primer. However, in some applications it has been found that good

(Continued on Page 94)

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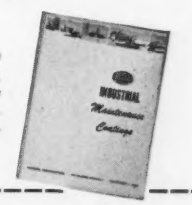

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Year's Activity Reviewd by St. Louis Section; Members Praised for Work on Conference

Newly elected officers for St. Louis Section are Charles W. Ambler, American Zinc, Lead and Smelting Co., chairman; O. W. Seibert, Monsanto Chemical Co., vice-chairman; Dean Jarboe, Dennis Chemical Co., secretary and A. O. Fisher, Monsanto Chemical Co., treasurer. Newly elected board members are: William C. Linton, Nooter Corp. and David E. Morris, Mallinckrodt Chemical Works. Results of the election were announced in a chairman's report, May, 1957 by George T. Shutt, Shutt Process Equipment Co., past chairman.

Mr. Shutt's comprehensive report also presented a resume of section activities for 1956-57. Most important single event in which the section participated was the holding of the 13th Annual NACE Conference in St. Louis with members of the section having important conference posts.

Members Are Praised

Success of the conference was due in large measure to St. Louis Section men, the report said, citing Otto Fenner, Monsanto Chemical Co., general conference chairman; W. O. Ries, Tretolite Co., local arrangements chairman; Walter B. Meyer, St. Louis Metallizing Co., exhibits co-chairman; Howard Nordquist, Jos. T. Ryerson & Sons, Inc., entertainment chairman; A. O. Fisher, technical program services chairman; E. L. Rice, Titanium Div., National Lead Co., registration chairman; Mrs. Mary Ellen Young, ladies entertainment chairman and Bob Harper, Nooter Corp., publicity chairman. Concurrent with the 13th Annual Conference and in cooperation with the national NACE, St. Louis Section sponsored a half hour TV program entitled "Corrosion and How It Affects You." The panel discussion, moderated by Lawrence E. Stout of Washington University, included W. A. Bonner of Shell Oil Co., E. F. Trunk, Laclede Gas Co., and N. E. Berry, Mallinckrodt Chemical Works.

One of the year's highlights in section affairs was the December meeting attended by William F. Fair, Jr., Koppers Co., Inc., then president of NACE. The meeting was a social one and included dancing and singing. It was suggested that such a meeting be planned on an annual basis. Dean Jarboe planned the meeting.

In addition to his conference work, the report stated, Bill Ries was instrumental in the reactivation of the North Central regional committee.

Duties Are Dispersed

During the year, an effort was made to apportion section duties and activities so that more persons were active on committees, rather than continuing it to a few board members as had been done previously. This system was recommended for future use. Committees and posts activated were: Historian, Otto Fenner; Bylaws, George Shutt; Membership, Robert Sanford, Nooter Corp.; Finance, Wallace Philoon, Jr., Mallinckrodt Chemical Works; Hospitality, Ed. Duce; Entertainment and Reservations, Dean Jarboe; Public Relations, Ed. Rice; Publications, George Purdy; Corrosion Directory Service, Charles Ambler.

Several suggestions were made by Chairman Shutt regarding section committee operation. These were: A committee be formed to establish standard procedures for operating the section and keeping records, with the possibility that once established, the procedures could be made part of the bylaws. The publication committee should be under the secretary and should inform members of NACE activities. A committee for corporate membership work should be formed.

Periods of service for all committeemen should not be concurrent so that continuity of membership on committees may be provided. Board meetings should include committee chairmen. Committees should be activated early in the season and efforts should be made to poll members as to their preference for committee work. Each committee should make known objectives to be attained during the year.

Bylaws of the section should be reviewed and revised with emphasis placed on election procedures. It was suggested that only one candidate for each office be named by the nominating committee with ample time and provision for other nominations by the general membership. Work to be done by the historical committee should be given high priority.

Deep Well Pump—

(Continued From Page 93)

results can be obtained without a primer.

Al Stetson discussed ceramic coatings of various types and uses. Porcelain enamels heretofore have required fusing at between 1450 and 1550 F. The high heats required tended to distort the mild steels used as a base, but new low firing colored enamels have been developed and are now being used on household appliances. New enamels for aluminum have been developed which are excellent for many applications where steel is not fully satisfactory.

Glass-Lined Containers Covered

Glass linings for water heaters, beer and wine vats are doing good jobs and are rapidly being improved. It is expected that glass linings performing satisfactorily at temperatures in the neighborhood of 180 F will be available shortly. Experiments are under way now on glass linings containing metal which will be more durable and less brittle. Jet engine parts are now receiving ceramic coatings for a variety of uses. New uses in this field and in missiles were described. Several slides were projected as illustrations.

Mr. Taitt spoke on new rubber compounds being used in aircraft, marine and industrial applications. These have good adhesion and good dielectric properties. They may be designed either for bonding metal to metal or other materials or for quick-stripping applications. Samples of the material were distributed showing bonding to wood, metal and concrete. The material makes excellent expansion joints. Also shown were air-operated guns for application in joints and fillets.

The annual social evening for members and their wives was planned for June 11 at the Marine Recruit Depot Officer's Club. Tentative speaker was Don Keller, district attorney.

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GROUND RESISTANCE—
SOIL RESISTIVITY
with
VIBROGROUNDS

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✓ Completely self-contained . . . dry-cell power supply ✓ Delivery promptly from stock ✓ Unaffected by stray A. C. or D. C. currents ✓ Simple push-button operation ✓ Rugged construction, steel case ✓ Accuracy within 3%

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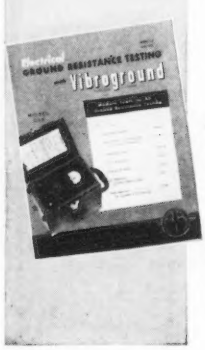
● FOR GROUND RESISTANCE TESTING ONLY: VIBROGROUND MODELS 251 and 255. Model 251 Ranges: 0-12.5, 0-125, 0-1,250 ohms. Model 255 Ranges: 0-3, 0-30, 0-300, 0-3000 ohms. Both Models: size 6" x 9" x 8½". Weight: 17 lbs. Price: Model 251, $120.50; Model 255, $142.50—F.O.B. Chicago.



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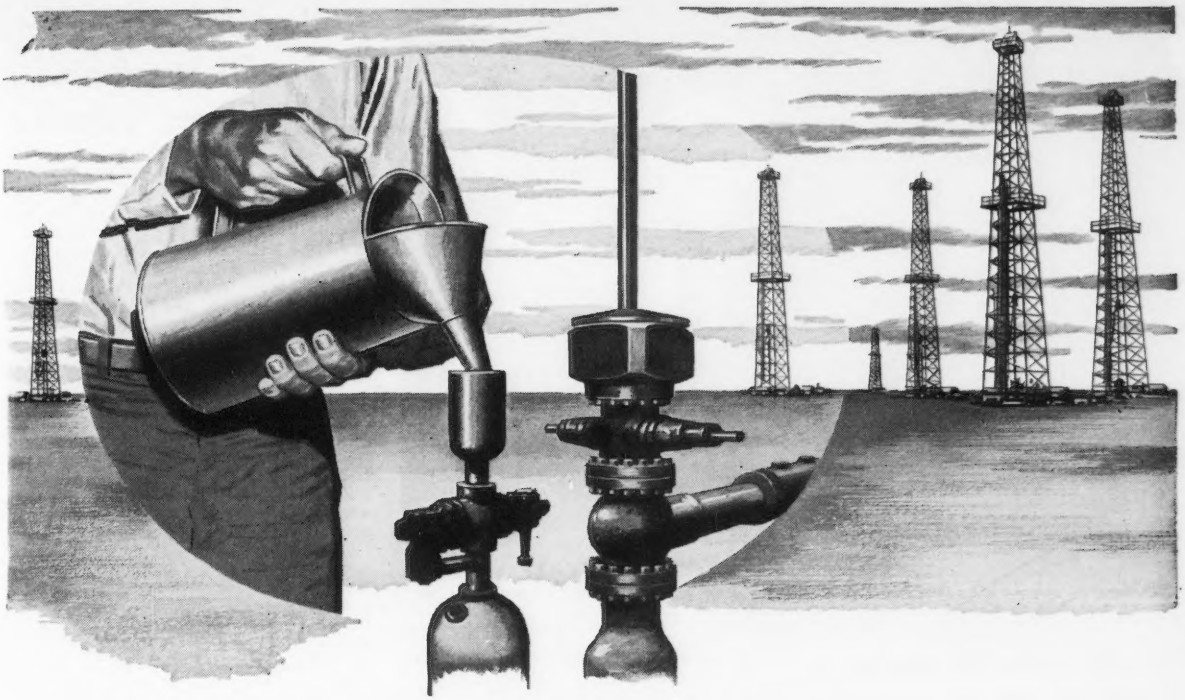

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AT PHILADELPHIA SECTION meeting May 10, J. Dwight Bird, The Dampney Co., Boston (left) presents a past chairman's certificate to Thomas F. Degnan, E. I. duPont de Nemours & Co., Inc., Wilmington.

Fair Discusses Membership Drive at Chicago Session

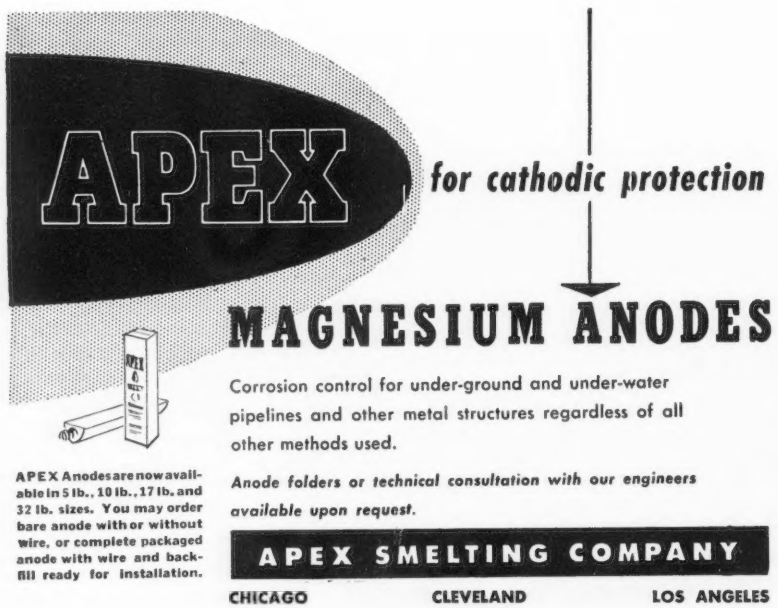
W. F. Fair, Jr., Koppers Co., Inc. immediate past president of NACE gave a brief talk at the May 21 meeting of Chicago Section in which he covered current membership in NACE and pointed out the current drive is to increase corporate membership. Dr. Fair discussed NACE Central Office's activity in fostering regional meetings and the national policies on regional exhibitions. He said the reorganization of the technical practice committees on industrial lines had proved beneficial.

Following Dr. Fair's presentation, a sound color film on the laying of a water pipe line for Colorado Springs was presented. The installation problem involved in laying this line emphasized Dr. Fair's closing comments.

During the meeting L. W. Ewing, past chairman of the Chicago Section presented the out-going chairman, R. B. Janota, a NACE lapel pin, engraved with his name and term of office. Such a pin is presented all chairmen of Chicago Section.

This was the final monthly meeting of the Chicago Section 1956-57 and was attended by approximately 53 members and guests.

The following officers were installed for the 1957-58 season: V. M. Kalhauge, Standard Oil Company of Indiana, chairman; H. C. Boone, Peoples Gas Light & Coke Company, vice-chairman; I. J. Acosta, Crane Company, secretary; and D. G. Keefe, Swift & Company, Treasurer.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APEX Anodes are now available in 5 lb., 10 lb., 17 lb. and 32 lb. sizes. You may order bare anode with or without wire, or complete packaged anode with wire and backfill ready for installation.

Philadelphia Section Hears Discussion of Glass Fibers, Epoxies

Marcus W. Keyes, manager of the Sales Engineering Department, Fiberglass Division, Pittsburgh Plate Glass Co., was the coffee speaker of the May 10 meeting of Philadelphia section at the Poor Richard Club. Mr. Keyes spoke on "Fibers of Glass with Properties that Make a New World of Products." Mr. Keyes described the manufacture of various types of glass fiber and illustrated the uses of each.

Walter A. Luce, supervisor of Non-Metallic Research and Development of The Duriron Company was assisted by John B. Kitteredge, in presenting a paper on "Cast Epoxy for Corrosion Application." Mr. Kitteredge discussed the chemistry of the epoxy resins and outlined the properties which made them useful for castings. Mr. Luce told of the actual castings made by The Duriron Company and gave mechanical strength, corrosion resistance and electrical properties.

During the business portion of the meeting, attended by 104 members and guests, J. Dwight Bird, chairman of Northeast Region, presented Tom Degnan, past chairman of the Philadelphia Section, a Past Chairman's Certificate for outstanding service.

Future meetings of the Philadelphia Section were announced as follows:
Tuesday, October 29, 1957—A panel discussion, "Materials of Construction."
Tuesday, December 17, 1957—Dr. M. G. Fontana, will discuss "Various Approaches to Corrosion Problems."

Pipe Line Coatings Covered In Shreveport Section Talk

"High Voltage Testing of Pipe Line Coatings," was discussed at the Shreveport Section meeting May 7 at the Washington-Youree Hotel. M. W. Belson, general manager of D. E. Stearns Co. was the speaker.

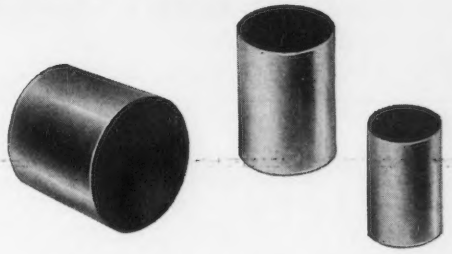
In tracing the development of commercial holiday detectors, Mr. Belson said that since their introduction, weight has been reduced from 300 to 12 pounds. Following a short period of discussion from the floor the meeting was adjourned.

Mr. Belson, an NACE member, was chairman of the Shreveport Section in 1948. He is co-author of a paper, "Technical Factors in Testing Pipe Line Coatings," presented at the South Central Region meeting and at the national meeting of NACE in 1949. Since 1949 he has been with the D. E. Stearns Co. as general manager. He served as an instructor at the first Appalachian Underground Corrosion Short Course in Morgantown, W. Virginia in 1956.

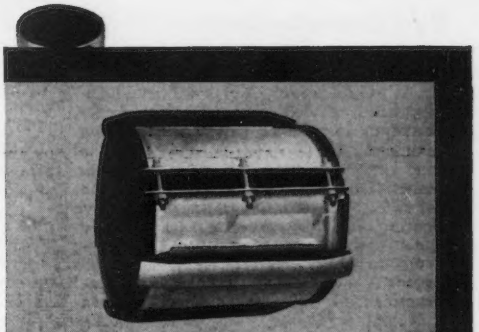
Instrumentation Is Topic At Corpus Christi Session

Approximately 35 members and guests attended the May 28 meeting of Corpus Christi Section. Frank I. Dorr, Jr., Dorr Engineering Company, Inc., spoke on Instrumentation. Among those present at the meeting was A. B. Campbell, executive secretary of NACE. The meeting was held in the Mirror Room of the Nueces Hotel.

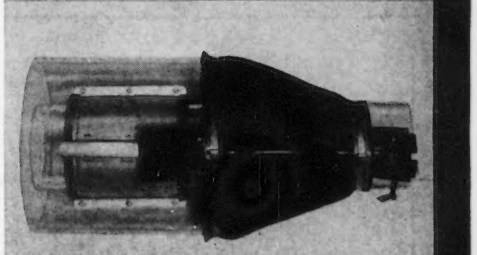
"Something From the Irishman"



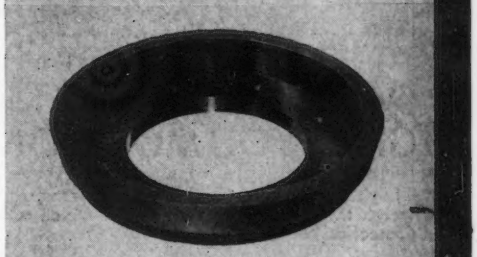
INSULATED COUPLINGS



PIPELINE CROSSING INSULATORS



PIPELINE CASING SEALS



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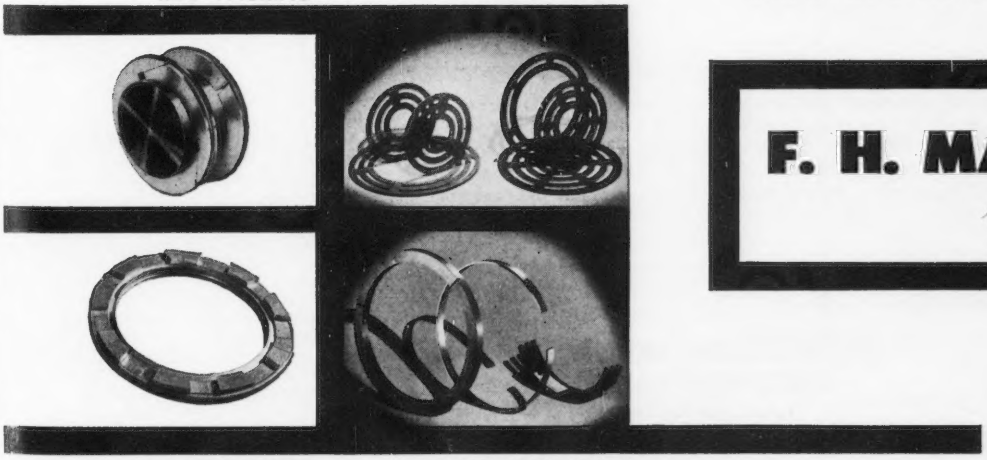

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W. G. Brander Is Elected By Edmonton Section

The following have been elected officers of the Edmonton Section for 1957-58: W. G. Brander, Northwestern Utilities, Limited, chairman; W. H. Seager, Canadian Protective Coating Ltd., vice-chairman; E. E. Moore, Interprovincial Pipe Line Company, secretary-treasurer. Committee chairmen are: Arrangements: G. E. Thompson, Canadian Protective Coating Ltd.; Membership: D. J. McQuarrie, Imperial Pipe Line Company; Program: J. L. Gattenmeyer, Canadian Protective Coating Ltd.; and Technical Practices: M. W. Clark, Canadian Chemical Company.

•

NACE now has 52 local sections.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1957

Oct. 1-4—North Central Region, Chicago, Sherman Hotel. Exhibition.

Oct. 1-4—South Central Region, Oklahoma City, municipal auditorium. Exhibition.

October 23-25—Western Region. Seventh Annual Conference, U. S. Grant Hotel, San Diego, Cal.

Oct. 23-25—7th Biennial Permian Basin Corrosion Tour. Midland-Odessa area, Texas.

Nov. 12-14—Northeast Region Fall meeting, Pittsburgh, Pa.. Penn-Sheraton Hotel.

1958

Mar. 17-21—NACE Annual Conference, Civic Auditorium, San Francisco, California.

October 5-8—Northeast Region, Somerset Hotel, Boston, Mass.

Oct. 15-17—North Central Region. Cincinnati, Ohio.

October 20-24—South Central Region. New Orleans, Roosevelt Hotel.

1959

NACE Annual Conference, Sherman Hotel, Chicago, Illinois.

SHORT COURSES 1958

October 22-23—Western Region. Corrosion Control Course (Evening Sessions) Hotel U. S. Grant, San Diego, Cal.

December 9-13—Biennial Short Course on Cathodic Protection. University of Illinois and NACE.

WHICH "HIGH-POTENTIAL" ANODE GIVES YOU MOST FOR YOUR MONEY?

They're both called "high-potential" magnesium anodes. They look alike. They weigh the same. How do you tell which one will give you the most cathodic protection for our money?

The answer is in their current efficiency—the useful ampere hours per pound of metal consumed—and their solution potential.

What can this mean to you? Assume a packaged, 17-lb. Galvomag—Dow's high-potential anode—would cost $9.95 plus $5.00 for installation. It operates at 50 percent efficiency (at a 0.1 ampere current drain) and has a life of 9.7 years. Other "high-potential" packaged anodes with a maximum efficiency of 30 percent have a maximum life of only 5.8 years. Operating at maximum efficiency, to give the same ampere-hour cost as the Galvomag, these anodes would have to sell for only $3.94!

As a service to the industry, CSI has found an independent laboratory that will determine the relative efficiency of anodes for you. The standard 14-day "pencil" test is used:

St. Louis Testing Laboratories
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This lab is 28 years old, and does work regularly for more than 2000 clients—including major oil, chemical and aviation firms. It is a member of the American Council of Independent Laboratories. (For more information, write C. D. Trowbridge, Director.)

Meanwhile, specify Galvomag, Dow's original high-potential magnesium anode, from CSI. You can be sure you are getting the most for your money—and exactly what you specify.

Write or call today. Quotations on cathodic protection materials or services without obligation. Ask for a free copy of the new CSI report on high-potential anodes in hot spot protection.

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Cleveland Hears Madison Discuss Epoxy Coatings

The final meeting of 1956-57 of Cleveland Section was held May 21, with approximately 65 members and guests present.

Ralph Madison, Technical Director of Truscon Laboratories spoke on "Epoxy Resin Protective Coatings." Colored slides were shown throughout Mr. Madison's talk depicting laboratory and field tests on epoxy resin coatings.

New officers for 1957-58 were elected as follows: A. R. Corlett, Harco Corporation, chairman; J. F. Bosich, Diamond Alkali Company, vice-chairman; and John Scott, Truscon Laboratories, secretary-treasurer.

Gleser Discusses Low Alloy Steels' Properties

Sol M. Gleser, A. M. Kinney, Inc., presented a paper on Corrosion Properties of Low Alloy Steel at the May 28 meeting of Southwestern Ohio Section attended by approximately 30 members and guests.

During the business portion of the meeting, Roy McDuffie, general chairman of the 1958 North Central Regional Meeting, discussed plans for the meeting and asked L. M. Lederer to make definite commitments of dates of October 15, 16 and 17, 1958.

An interesting question and answer session followed the presentation of Mr. Gleser's paper.

Bumpus Talks at Detroit

Herbert Bumpus of the Michigan Bell Telephone Company presented a demonstration and lecture on Microwaves, Transistors and the Solar Battery at the May 16 meeting of Detroit Section. The meeting, held in the Engineering Society Building of Detroit was attended by 11 members and 6 guests.

•

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Concentric Completion Corrosion Is Discussed

Approximately 40 members and guests attended the May 9 meeting of Teche Section at the Petroleum Club in Lafayette, Louisiana and heard Lee Roy De Rouen, of the Union Oil and Gas Corporation of Louisiana speak on "Corrosion Control of Concentric Completions."

Plans for the Corrosion Short Course to be held in June were completed. This was the last section meeting of the current season. Meetings will be resumed in the fall.

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NACE now has 52 local sections.

French Engineers Attend Tulsa Section Meeting

Louis M. Cauchois, Societe Nationale des Petroles d'Aquitaine, Paris, France and J. R. Callais, of Pau, France, near the Lacq field were guests of Tulsa Section at its May 27 meeting.

Robert Goodnight, Pan American Petroleum Corp. Research Center spoke on Special Testing Techniques for Protective Coatings. Fifty-two persons were present.

Next meeting of the section is scheduled September 23.

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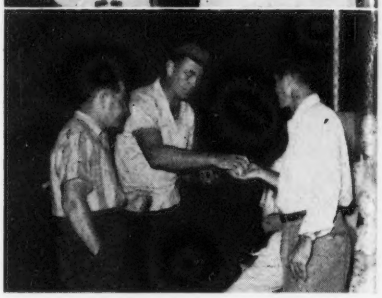
Abstracts in CORROSION come from 28 agencies.



AT HOUSTON SECTION MAY PICNIC, top, left to right, First Assistant Cook Joe L. Mitchell, United Gas Co. and Chief Cook, C. B. Tinsley, Bell Telephone Company talk over barbecue business. Bottom, Ernie Utzler looks on while Charles A. Nehring, Pittsburgh Plate Glass Co. (center) hands out a prize to golfer Patrick R. Donnelly, Tretolite Co.

Shreveport and Houston Schedule Summer Meetings

Shreveport and Houston Sections have announced intentions to continue monthly meetings during the remainder of the summer. These sections are the only two that apparently plan to continue meetings. The remainder of NACE's sections have scheduled activities for the fall beginning with September, October or November.

In the meanwhile, considerable time is being spent by sponsoring sections on regional meetings, reports on which are expected to appear during the summer in issues of CORROSION.

Holloman ADC Seeks Physical Metallurgist

Holloman Air Development Center, Holloman Air Force Base, New Mexico is seeking a physical metallurgist, GS-1321-7, annual salary $5335. Persons interested are invited to write to: Civilian Personnel Office at the air force base.

Nondestructive Test Papers To Be Published by ASTM

Forty papers on nondestructive testing methods released by the Atomic Energy Commission at an April 16-18 meeting in Chicago will be published by American Society for Testing Materials.

•

CORROSION publishes annually in December an alphabetical subject index to its Technical Section.

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of new vinyl primer

New PRUFCOAT VINYL-WELD PRIMER P-62 has a "formulated-in" safety factor of *damp surface tolerance* that gives you low-cost insurance against costly premature coating breakdowns due to surface dampness at the time of primer application.

The above photograph shows what happened . . . just a few short weeks after job completion . . . when an ordinary vinyl primer was used over damp, wire-brushed steel. Failures of this kind can play havoc with painting maintenance costs . . . *cut sharply and unexpectedly into your net operating profit.*

In money-saving contrast, PRUFCOAT VINYL-WELD PRIMER P-62 . . . under identical damp surface application conditions . . . provides *complete protection* with no sign of breakdown after many months of punishing corrosive exposure.

But this damp surface safety factor is by no means all this new primer has to offer. There are these other important primer features too . . .

SUPERB ADHESION • SUPERIOR RUST INHIBITION
EXCELLENT CHEMICAL RESISTANCE • QUICK DRYING

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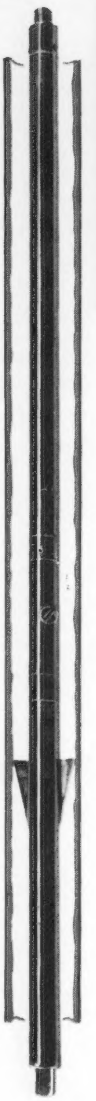

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Shut-in periods are slashed when this liquid corrosion inhibitor dump bailer is used in producing oil or gas condensate wells. Average time per 8,000 to 9,000 ft. is only one hour. This saving in time *cuts labor costs* to a *new low* for corrosion inhibitor treatment. Easily lowered from conventional wire line.

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By using *concentrated* liquid inhibitor, it forms a *superior* protective film as it moves up the string. Five quarts of inhibitor are *all* that are required to protect the average well for a month or longer.

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TECHE SECTION Short Course committeemen: left to right, standing, LeRoy DeRouen, Union Oil and Gas Corp. of La., afternoon session chairman; Bill Paine, Southwestern Louisiana Institute, general planning committee; seated, Max I. Suchanek, Dowell, Inc., director; Frank Shofner, Aquaness Corp., Planning and Program Committee; Nathan James, Texas Company and F. L. Rhodes, Otis Pressure Control, Inc., Speakers Committee; Phil Brook, Dowell, Inc., Entertainment; Wayne Grant, Texas Co., Registration; Vernon Sibille, Superior Oil Co. and W. A. Odom, Tretolite Co., Housing and Meals.

Current Corrosion Questions Topic of N. Texas Section

"Current Questions in Corrosion" was the subject for group discussion at the North Texas Section meeting May 6 at the Torch Restaurant, Dallas. All members attending were invited to contribute to the discussion. Many members expressed approval of the new audience participation program.

The questions discussed were: Can customary criteria of gas well corrosion be used with super high pressure wells? How can corrosion inhibitors be used in water injection systems? How can Dallas water best be handled in air conditioners? What is the economical balance between coating and cathodic protection?

Leaders in the discussion were: Morris Bock, Sun Oil Co.; Jerry R. McIntyre, The Atlantic Refining Co.; Floyd Blount, Magnolia Petroleum Co.; H. L. Bilhartz, Production Profits, Inc.; Charles D. Holland, Jr., Sun Oil Co.; and R. B. Bender, Plastic Engineering & Sales Corp.

Next meeting of the section will be in September.

Cathodic Protection of Pipe Lines Is Shreveport Topic

Shreveport Section heard William F. Levert, senior corrosion engineer for the United Gas Pipe Line Co., Shreveport, La. speak on "Cathodic Protection of Underground Pipe Lines" at its June 4 meeting in the Washington-Youree Hotel.

Mr. Levert presented some of the generally established data regarding electrical protection of underground pipe lines. Certain factors of installation and maintenance should be considered in detail and with great care whenever a cathodic protection system is planned, he said. Mr. Levert is Assistant Secretary-Treasurer of the South Central Region, NACE and trustee of Shreveport Section.

M. J. Olive, Arkansas Fuel Oil Corp., was appointed chairman to provide entertainment for the members and their lady guests for the July 2 meeting at the Washington-Youree Hotel.

Teche Registrants Favor Three-Day 1958 Short Course

A three-day short course on corrosion control was favored by 75 percent of those attending the June 6-7 Teche Section Short Course on Corrosion at Lafayette, La. Max I. Suchanek, Dowell, Inc., course director reported those queried about repeating the course during 1958 favored sessions on plant corrosion and oil and gas field corrosion work.

Of the 121 persons registered, including 11 speakers, 91 came from Louisiana, 15 from Texas, and one each from Arkansas, Mississippi, Missouri and three from Oklahoma.

East Texas Section Hears Discussion on Coatings

R. M. Ives, Jr., Humble Oil and Refining Company, Houston, spoke on Protective Coatings for Oil Field Use at the May 28 meeting of East Texas Section. Approximately 45 members and guests attended. Mr. Ives gave an interesting talk on ordinary painting for protection against weather and on special coatings and application methods for protection against high temperature, abnormal moisture and chemical corrosion. His talk was illustrated with color slides, showing examples of various coatings.

B. G. Hustead, East Texas Salt Water Disposal Company was elected to serve the unexpired term of vice-chairman.

It was announced the next meeting of the section will be September 24.

Harry T. Paterson

Harry T. Paterson, 46, operational manager of the Harbor Island and Kure Beach Corrosion Test Stations of The International Nickel Co., Inc. died suddenly at his home at 823 Windsor Drive, Wilmington on May 27. He had been with Inco since 1946.

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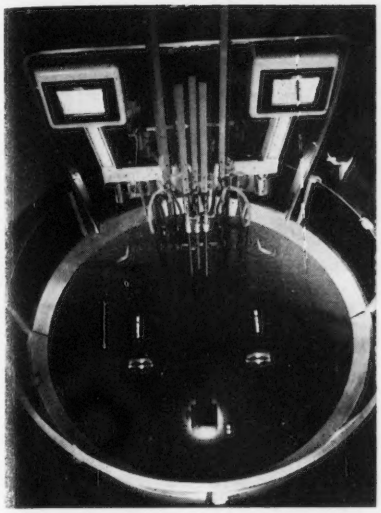

THE "GENEVA REACTOR" is shown here as it looked when erected in Geneva, Switzerland. Francois Kertesz, at right, described the project to the East Tennessee Section in a recent meeting. The reactor was the first to be built by the United States on foreign soil.

McLaren Is Elected Chairman by East Tennessee Section

East Tennessee Section has elected new officers for the 1957-'58 term. James A. McLaren, Oak Ridge National Laboratory, Division of Union Carbide Corporation is the new chairman. Solon Walker, East Tennessee Natural Gas Co., Knoxville, Tenn., was re-elected vice-chairman and Frank A. Knox, Oak Ridge National Laboratory, Union Carbide Nuclear Co., Oak Ridge, Tenn., secretary-treasurer. Outgoing chairman was DeWitt D. Stone, The Duriron Co., Inc., Nnoxville, Tenn.

Results of the election were announced at the April 29 meeting in the Frontier Room, Bill's Restaurant, Oak Ridge, Tenn. Francois Kertesz, Oak Ridge National Laboratory, Union Carbide Nuclear Co., spoke on the "Geneva Reactor." He told the group of the problems, both serious and humorous, in erecting in Geneva, Switzerland, the first United States reactor to be built in a foreign land.

Tank Trimmed Level

In excavating for the swimming pool type reactor, archaeological ruins, thought to be ancient Roman, were uncovered. Another problem had to be solved by the ceramics personnel in the materials of the reactor. Then in lowering the tank, the cables broke. The dropped tank did not break but fell slightly off the perpendicular. Solution to this problem was to level the top by cutting the rim, leaving the tank at an angle which was virtually unnoticeable.

Problems of a less technical nature also arose. One was the instruction of the attractive European girls who with but little experience as physicists acted as guides in explaining the operation of the reactor in German, Spanish, French

(Continued on Page 104)

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ASTM Elects LaQue

F. L. LaQue, Manager of the Research and Development Division, The International Nickel Co., Inc., New York and formerly president of the National Association of Corrosion Engineers has been elected to a two-year term as vice-president of American Society for Testing Materials.

McLaren Is Elected—

(Continued From Page 103)

and English. Another development was the "wine break" of Swiss mechanics to accompany the "coffee break" of the Americans. Some persons made both "breaks," according to Mr. Kertesz.

Three Meetings Are Held

During 1956 the Section held three meetings. A joint meeting with the Society for Non-destructive Testing was held March 7. Peter K. Bloch, Branson Instruments, Inc., spoke on "Topic, Theory and Application of Ultrasonic Resonance Test Instruments." The meeting was held at the Oak Ridge Institute of Nuclear Studies, Lecture Room.

At the May 21 meeting, J. E. Draley, Argonne National Laboratory spoke on "Some Unusual Effects of Hydrogen in Aqueous Corrosion." The meeting was held at Bradshaw's Steak House, Oak Ridge Highway.

A joint meeting with the American Society for Metals was held in October. The speaker was R. A. Belser, associate professor at Georgia Technological Institute. His topic was "Oxidation Resistant Coatings and Metal Coating Techniques." The meeting was held at the Knights of Columbus Hall, Oak Ridge, Tenn.

Bialosky

Erickson

Phelps

Renshaw

Ringer

Golden Triangle Meeting Symposia Chairmen Named

The following have been named chairmen of the indicated symposia to be held during the November 12-14 Golden Triangle Meeting of Northeast Region at Pittsburgh. Jerome Bialosky, Kopper Co., Inc., Pittsburgh, is technical program chairman.

Underground Corrosion Symposium, C. A. Erickson, Peoples Natural Gas Co., Pittsburgh.

Chemical Industry, Howard Trusler, Pittsburgh Coke & Chemical Co., New York.

Protective Coatings, George Seagren, Mellon Institute, Pittsburgh.

New Metallurgical Developments, Herbert Phelps, United States Steel Corp., Applied Research Laboratory, Monroeville, Pa.

Power Industry, Joseph Cameron, Elliott Corp., Jeanette, Pa.

Instruments for Corrosion, Francis W. Ringer, Narberth, Pa.

W. E. Renshaw, Allegheny Ludlum Steel Corp., Brackenridge, Pa. is chairman of the plant tours committee.

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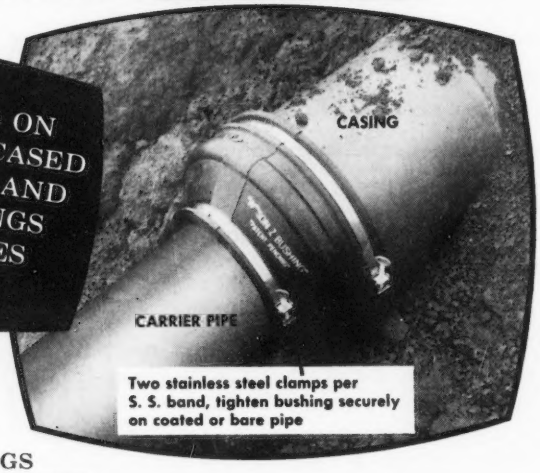

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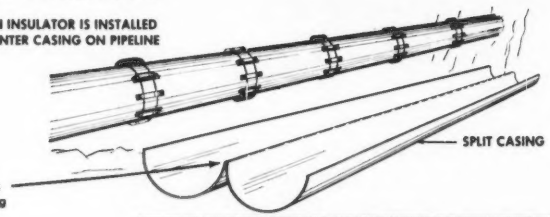

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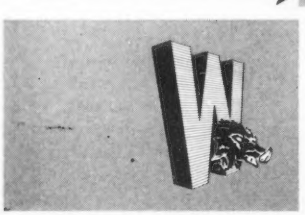

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1957 YEARBOOK
National Association of Corrosion Engineers
1061 M & M Building, Houston 2, Texas
Publication 57-16
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NACE 1957 Yearbook Copies Are Available

The 1957 NACE Yearbook, Publication 57-16, containing listings of members geographically and alphabetically has been published and is available for sale now. Only members of NACE are authorized to purchase copies at $2.50 each. Orders, accompanied by remittances payable to NACE should be forwarded to NACE Central Office, 1061 M & M Bldg., Houston.

The yearbook also includes a committee directory, the technical committee operations manual, a short history of the association, a description of awards and names of recipients and articles of organization and by-laws.

By action of the NACE board, it was decided copies should be made available for sale to members for any number of copies they may desire at $2.50 per copy and that gratis copies would be made available only to national, regional and sectional officers of the association.

The yearbook was issued last in 1955.

John R. Culver Named to Transportation Committee

John R. Culver, Coulter Steel and Forge Co., Emeryville, Cal. has been named head of the Transportation Committee for the San Francisco 1958 NACE Conference and Exhibition. Mr. Coulter, a 1940 graduate of the University of California in petroleum engineering, is a sales engineer.

Culver

The Exhibits Committee is working on details of the displays of materials and equipment and a budget has been prepared for presentation to the NACE board of directors.

San Joaquin Section Sets Topics of Future Programs

A series of future programs dealing with corrosion in oil production, processing and gas transmission and agricultural and metallurgical aspects of corrosion in the San Joaquin Valley are scheduled for San Joaquin Section.

At a June 19 meeting the scheduled program included showing the International Nickle Company motion picture "Corrosion in Action," by Ed Leduc, Dearborn Chemical Co. and a discussion of general oilfield corrosion by Preston Hill, Signal Oil and Gas Co.

Water Corrosion Test Paper

Experiences With Corrosion Testing in Condensate Systems, a technical paper by E. A. Yorkgitis, Hagan Chemicals and Controls, Inc. was given during the June 3-6 National District Heating Association Convention at Hot Springs, Va.

HYDROGEN ABSORPTION, EMBRITTLEMENT and FRACTURE of STEEL

By Arnold E. Schuetz, Research Department, Bridgeport Brass Company, Bridgeport, Conn. and W. D. Robertson, Hammond Metallurgical Laboratory, Yale University, New Haven, Conn.

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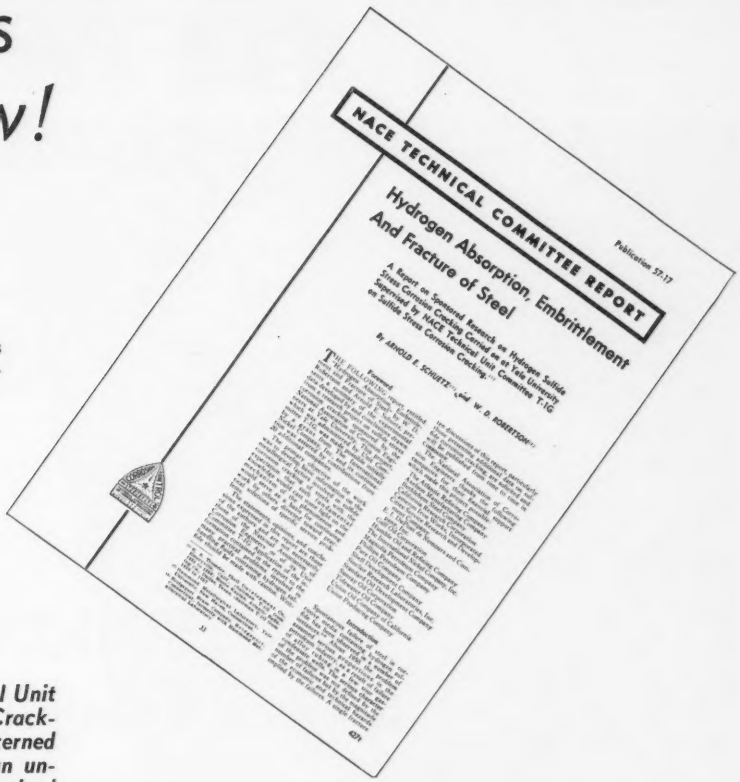

This 24-page illustrated report is replete with figures and tables on this basic research project's findings.

Basic Facts on Mechanism of Hydrogen Sulfide Stress Corrosion

The findings of a 4-year inquiry into the fundamental reactions involved in the so-called spontaneous fracture of steel as a result of hydrogen absorption in the presence of sulfides.

This project, supervised by NACE's Technical Unit Committee T-1G on Sulfide Stress Corrosion Cracking, was financed by industrial firms concerned with this problem in the expectation that an understanding of the fundamental reactions involved would lead to a solution.

Data in this report are expected to be useful not only in immediate steps to reduce the losses due to spontaneous failure but also as a foundation for subsequent inquiries into phenomena associated with hydrogen absorption and embrittlement of steel.

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1956 CORROSION CONTROL REPORTS TO ISCC

Growing Interest In Corrosion Is World Wide

Reports of organizations affiliated with the Inter Society Corrosion Committee indicate the high level of interest in corrosion control is increasing in North America and abroad. Widespread interest is evidenced in research projects into matters of both practical and theoretical interest.

The number of technical meetings at which discussions on corrosion control take place and the growing number of organizations with corrosion control functions and interests substantiate the view that corrosion mitigation is of pressing importance. New groups are being formed overseas at a rapid rate and increased activity among existing groups is reported.

It is safe to predict that in the foreseeable future no country with any appreciable amount of industrial plant will be without either a formal or informal corrosion investigation organization.

Reports on 1956 activities on ISCC affiliates follow:

AMERICAN CHEMICAL SOCIETY—Delegates: Aaron Wachter, Shell Development Co., Emeryville, Cal. and Norman Hackerman, University of Texas, Austin.

ACS sponsored one symposium on corrosion in the petroleum industry at its Dallas spring meeting. A number of papers on corrosion appeared in the society's journals during the year.

Hackerman

Compton

Officers of the Inter Society Corrosion Committee are Norman Hackerman, University of Texas, Austin and K. G. Compton, Bell Telephone Laboratories, Murray Hill, N. J., chairman and vice-chairman.

AMERICAN ELECTROPLATERS' SOCIETY—Delegate: W. A. Wesley, International Nickel Co., Inc., Bayonne, N. J.

The following AES Projects relate to corrosion:

Project 5—Effects of Impurities in Nickel Solutions. Effect of aluminum in thin deposits erratic; in heavy deposits insignificant up to "saturation." Manganese had no effect up to 300 ppm and calcium up to about 700 ppm.

Project 12—Continues to study effects of adhesion and corrosion of electrodeposit caused by oxide films on copper.

Project 13—Recently resumed. Measuring dimensions of pores at various levels in deposit.

Project 14—Studying effects of basis metal defects on corrosion protection of electrodeposits. Correlation is definite between basis metal inclusions and scratches and corrosion protection of thin deposits. Non-conducting silica is worst offender.

Project 15—Investigating reproducibility of accelerated corrosion tests. The acetic acid spray test and Corodkote test are under consideration now.

AMERICAN INSTITUTE OF CHEMICAL ENGINEERS—Delegates: H. F. McConomy, Atlantic Refining Co., Philadelphia and R. R. Pierce, Pennsalt Chemicals, Inc., Philadelphia.

A manual on corrosion testing is being prepared by a subcommittee of the Equipment Testing Procedures Committee. Four papers on corrosion subjects appeared in Chemical Engineering Progress.

AMERICAN SOCIETY FOR METALS—Delegates: M. G. Fontana, Ohio State University, Columbus; R. B. Mears, U. S. Steel Corp., Monroeville, Pa.

Twenty-two papers on various aspects of metal corrosion were published during the year, including one on thorium and high-thorium alloys.

Sixteen chapter meetings had programs at which corrosion control matters were discussed. These included a corrosion symposium at Louisville Chapter involving five speakers.

ASM Review of Metal Literature published 550 abstracts on corrosion during the year.

AMERICAN SOCIETY FOR TESTING MATERIALS—Delegates: F. L. LaQue, The International Nickel Co., Inc. and A. W. Tracy, American Brass Co., Waterbury, Conn.

Jerome Strauss, Vanadium Corp. of America, New York, N. Y. is chairman of the Advisory Committee on Corrosion which is responsible to the ASTM board and operates the ASTM test sites in the United States and Canada. A new test site in New Jersey will replace the present one on top of the port of New York Authority Building.

Complimentary copies of the 1956 ACC report are available from ASTM headquarters, 1961 Race St., Philadelphia 3, Pa.

Short reports on current committee activities follow:

Committee A-5 on Corrosion of Iron and Steel

New Tentative Specifications for Zinc-Coated (Galvanized) Steel Poultry Netting (Hexagonal and Straight line) and Woven Steel Poultry Fencing (A390-56T).

The committee is currently carrying out a collaborative test program using hand-held magnetic gages. These gages use magnetic flux as a means of determining thickness of metallic coatings.

The committee has made the 20-year

(Continued on Page 109)

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Growing Interest—

(Continued From Page 108)

inspection of the wire and fencing at nine ASTM atmospheric exposure test sites. There data are now being assembled. The committee will in the near future publish this 20-year information together with data and photo-micrographs from the 1939 wire test report of Subcommittee VIII on Field Tests of Metallic Coatings.

Atmospheric exposure of hardware and plate specimens at four ASTM exposure test sites is planned. The specimens will include hot-dipped and sprayed zinc and aluminum on carbon and alloy steels.

Committee A-7 on Malleable-Iron Castings

An atmospheric exposure program involving 1560 specimens exposed at five ASTM exposure test sites is being assembled. The specimens include various types of malleable, nodular, and mild steel.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys

The committee is assembling stainless steel materials in the form of tubing, wire and coil springs. Specimens will be exposed at four ASTM atmospheric exposure test sites. The program is expected to be ready by March, 1958.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys

The committee has collected the assemblies of stainless steel disks coupled to other metals which have been on exposure for 15 years. The data to be published from the exposure test will include contact resistance and after cleaning, visual changes and loss in weight.

The committee recently has completed gathering data to determine the degree of interest in a new test program embracing metals and alloys that have become of commercial importance in the last 25 years. As a result of these inquiries, offers have been received indicating cooperation in supplying specimens of the following metals and alloys: 16 aluminum alloys, 18 copper alloys, lead (6 percent antimony), 4 magnesium alloys, 2 molybdenum alloys, 5 nickel alloys, commercial grade tantalum, 8 titanium alloys, 2 zinc alloys and commercial grade zirconium. Consideration may be given to the inclusion of super purity aluminum (99.99 percent min.), beryllium alloys and Zircaloy-2.

Details to be developed from this program include exposure at four ASTM test sites, test periods up to 20 years, tests for thickness, weight change, tensile strength, pit depth and chemical constitution.

The committee would appreciate comments and suggestions which will enhance the value obtained from the new exposure program.

Committee B-7 on Light Metals and Alloys, Cast and Wrought

Specimens of 27 aluminum and 11 magnesium alloys, including welded and riveted specimens, exposed for three years have been examined and tested. This evaluation program has as its objectives the study of new alloys appearing in ASTM specifications, the study of fabricated specimens used by industry and a study of both permanent mold and sand casting alloys. The result of this inspection will be published in the Committee B-7 Annual Report.

Committee B-8 on Electrodeposited Metallic Coatings

A new exposure test program will shortly begin in which a number of cadmium plated steel panels will be exposed to test supplemental chromate films. A fraction of the test panels will be provided with buttons of dissimilar metals for study of galvanic action as influenced by the chromate film. The base metals will include: brass, copper, stainless steel and a proprietary magnesium and aluminum alloy. These specimens will be placed for exposure this spring.

Committee D-15 on Engine Antifreezes

This committee has published a method for glassware corrosion test for engine

(Continued on Page 110)

Meeting Dates Change Is Considered by ISCC

Members of the Inter Society Corrosion Committee are being asked to decide on a proposal calling for a change in the committee's schedule of meetings. Because of conflicts during national meetings and because some members believe meetings on a regional basis would have more interest and better attendance, the problem has been put on letter ballot for a decision.

The Subcommittee on Standards Terminology has prepared List No. 1—Glossary of Corrosion Terms and submitted it for publication.

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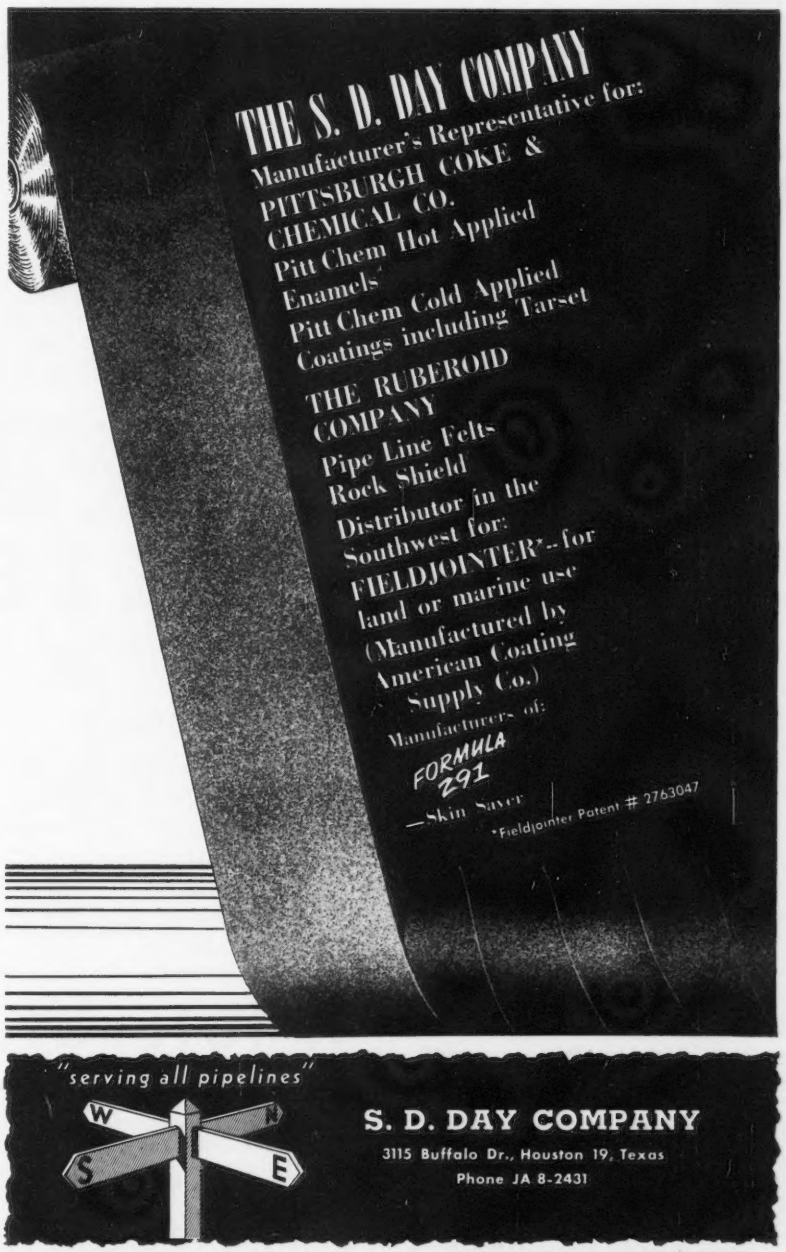

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Growing Interest—

(Continued From Page 109)

antifreezes (ASTM designation: D 1384-55T). The committee is working currently on a bench-type test for determining corrosion in a full scale engine block.

Committee D-19 on Industrial Water

The committee is considering a program of cooling water corrosion testing using metal coupons. Committee D-19 would like to solicit interest or experience in the use of coupons for this proposed program.

AMERICAN SOCIETY OF MECHANICAL ENGINEERS—Delegate: E. P. Partridge, Hagan Corp.

Four of the research committees of the American Society of Mechanical Engineers are either now engaged in or are planning research projects dealing with corrosion.

Effect of Temperature on the Properties of Metals (Joint ASTM-ASME Committee)

A project which will evaluate the resistance of low alloy ferritic steels to oxidation by steam, air and gas at elevated temperatures is being conducted by the Steam Power Panel in cooperation with steel manufacturers and power plants.

Boiler Feedwater Studies

The Corrosion Subcommittee is preparing a prospectus on controlled dynamic studies of the corrosion of iron by water and on the corrosion of copper and copper alloys by water. Data presently are lacking for most of the range of temperatures encountered in contemporary steam power plants.

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High Temperature Steam Generation

During 1956, a test facility to which the U. S. Naval Engineering Experiment Station contributed largely in design, fabrication and assembly was installed in the Philip Sporn Plant of American Gas & Electric Service Corporation. This test facility will be operated for three years during which time specimens will be removed at intervals of six, 12, 18, 24 and 36 months after exposure to steam at temperature levels of 1100, 1200, 1350 and 1500 F respectively.

Further development work has been completed on equipment at Purdue University for measuring the thermal conductivity of scale formed on the inside surface of the specimens.

A 20-minute, 16-mm sound motion picture in color has been completed by Wilson F. Erskine, Project Manager, as a pictorial record of construction details on methods of assembly of components. This picture is available for use by sponsors and other interested groups.

Corrosion and Deposits from Combustion Gases

Funds are being solicited for a research program on the selection and critical examination of available information on corrosion and deposits from combustion gases.

AMERICAN SOCIETY OF REFRIGERATING ENGINEERS—Delegate: Richard A. Line, Hussmann Refrigerator Co., St. Louis, Mo.

ASRE members indicated an increasing interest in corrosion problems and their solutions in 1956. Increased sales of room air conditioners and the use of cooling towers, evaporative condensers and outdoor air cooled condensers have emphasized the need for thorough knowledge of all known corrosion preventive measures.

A corrosion committee, consisting of the following men have been appointed to carry on activities to assist in corrosion activity communications:

During the 1956 calendar year, ASRE sponsored the following activities: Spring National Meeting, Cincinnati: Forum, June 5, 1956—Corrosion of Air Conditioning Blower Equipment. Winter Meeting, Boston: Forum, November 27, 1956—New Developments in Corrosion Preventive Finishes.

ASRE Research Committee: Appointed a task committee to study the effects of contaminants (air, moisture, etc.) inside a refrigeration system. This committee has been active and is scheduled to make a preliminary report at the spring meeting in June, 1957. Its report will deal with internal corrosion in addition to other factors.

ASRE Data Book: During 1956 it was decided to add a chapter to the Data Book. This chapter will deal with Corrosion and Water Treatment—Causes and Control.

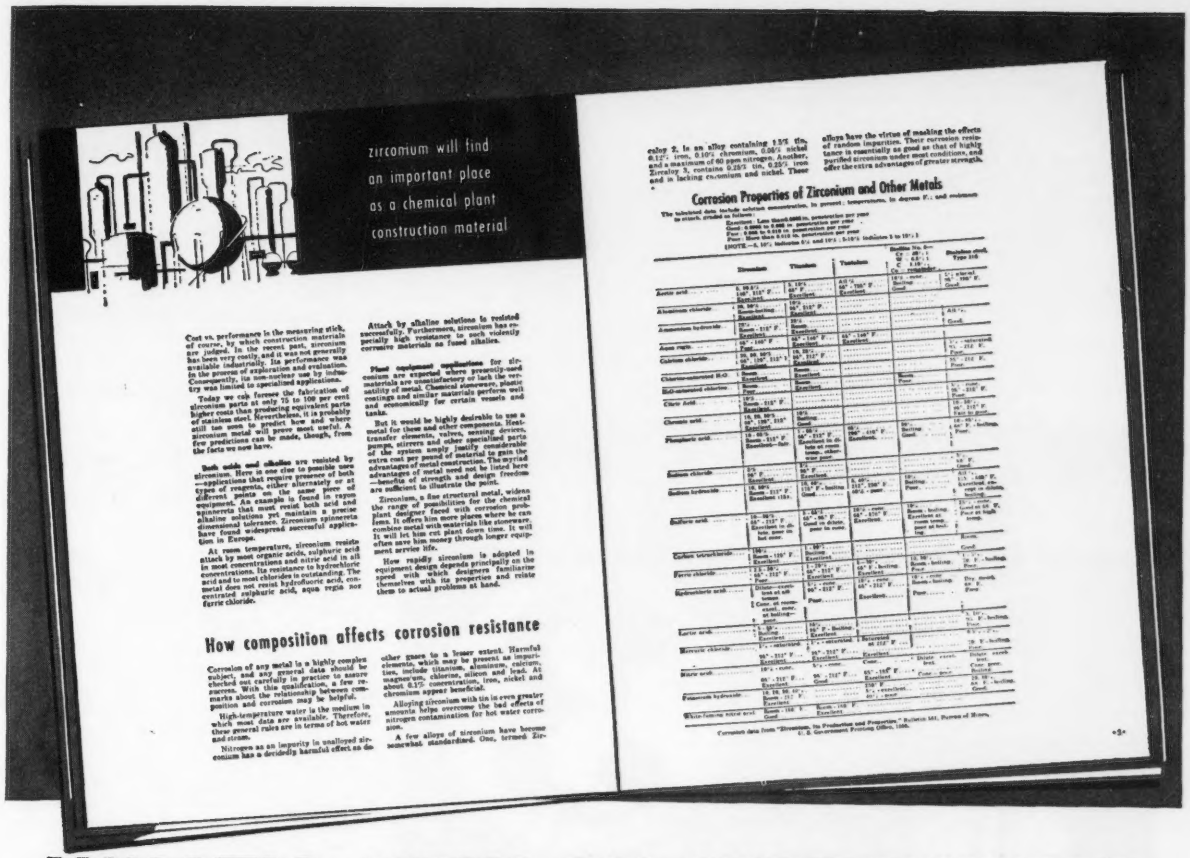
All corrosion activities have been well attended and future activities appear to be of even greater interest.

AMERICAN WELDING SOCIETY—Delegates: M. A. Scheil, A. O. Smith Corp., Milwaukee; W. H. Watkins, Metallizing Co. of America, New York.

Report of the Welding Research Council—High Alloys Committee, W. H. Binder, chairman:

For the past four years, a study of

(Continued on Page 112)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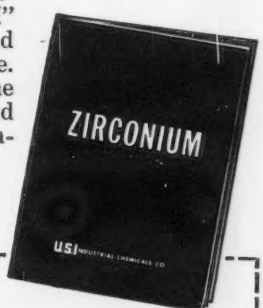

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Growing Interest–

(Continued From Page 110)

the effect of weld metal composition on the properties of Type 347 steel welds has been conducted by the Arcos Corporation for the AEC. This program was outlined initially by the High Alloys Committee, W.R.C. Advisory Subcommittee on the Welding of Stainless Steels. This contract has been renewed for another year and the extension will permit a study of high temperature properties of Type 347 steel weld metal. Major object of this program is to determine the ability of fully austenitic compositions to resist cracking and embrittlement and to determine their suitability for service. A paper covering the portion of the program devoted to the evaluation of the electrodes to meet standard fabrication codes and the determination of tensile and impact properties, entitled "Mechanical Properties of Modified Type 347 Weld Metals," was presented before the A.W.S. in October.

The High Alloys Committee, through its Field Corrosion Subcommittee, is conducting a study of the corrosion resistance of austenitic steel weldments. This program has progressed to the point that samples, 12 by 12 inches in size, comprising one-quarter of the total program, have been welded and heat-treated. The heat-treatment of the squares was conducted by the Armco Steel Corporation and the Welding by the Arcos Corporation. To date, sixteen sets, involving a total of 256 test specimens, have been machined and are being prepared for plant exposure tests. The machining of the final specimens is being carried out by Combusting Engineering, Inc., Chattanooga Division, and the preparation required for plant exposure tests is being performed through the co-operation of the International Nickel Company. It is hoped that the entire set of specimens comprising the first quarter of the test program will be available for testing in the near future.

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ASSOCIATION OF AMERICAN RAILROADS—Delegates: G. M. Magee and W. M. Keller, AAR, Chicago.

The following corrosion interests were pursued at the Central Research Laboratories of the Association of American Railroads:

Engineering Division

Evaluation of corrosion inhibitors for use in ice-salt mixtures in refrigerator cars.

Development of various screening techniques such as weight loss studies, changes in potential, etc.

Construction of a humidity cabinet in which relative humidity will be varied constantly through the range 100 to 20 percent.

Mechanical Division

The program for evaluating protective coatings for application to railroad "truck" side frames was continued. A report was prepared showing the relative effectiveness of the coatings studied in preventing corrosion of the side frame. The relative effectiveness is based on laboratory procedures designed to simulate the conditions which might exist in service. The recommendation has been made that several coatings be applied to truck side frames in order to correlate the laboratory tests with service performance. If correlation can be established, other coatings will be tested for possible application in the side frame corrosion problem.

ELECTROCHEMICAL SOCIETY, INC.—Delegates: H. H. Uhlig, Massachusetts Institute of Technology, Cambridge; K. G. Compton, Bell Telephone Laboratories, Murray Hill, N. J.

The Corrosion Division of the Electrochemical Society held its last symposium in Cleveland last October and will hold its next meeting in Buffalo, New York, October 6-10, 1957. Present officers of the division are: J. E. Draley, chairman, C. V. King, vice-chairman, M. A. Streicher, secretary-treasurer.

The society, cooperating with the Bunsen Gesellschaft and the Faraday Society, is arranging an International Symposium on Passivity to be held near Darmstadt, Germany, September 2-7, 1957. Papers presented at this meeting will be published in a special issue of the Zeit. fur Elektrochemie.

FEDERATION OF PAINT AND VARNISH PRODUCTION CLUBS—Delegate: G. Schurr, Chicago.

James W. Tomecko, now president-elect of the FPVPC has a special assignment to chart a course for future corrosion research to be supported by the Federation.

A project designed to determine minimum paint film thickness for structural

(Continued on Page 114)

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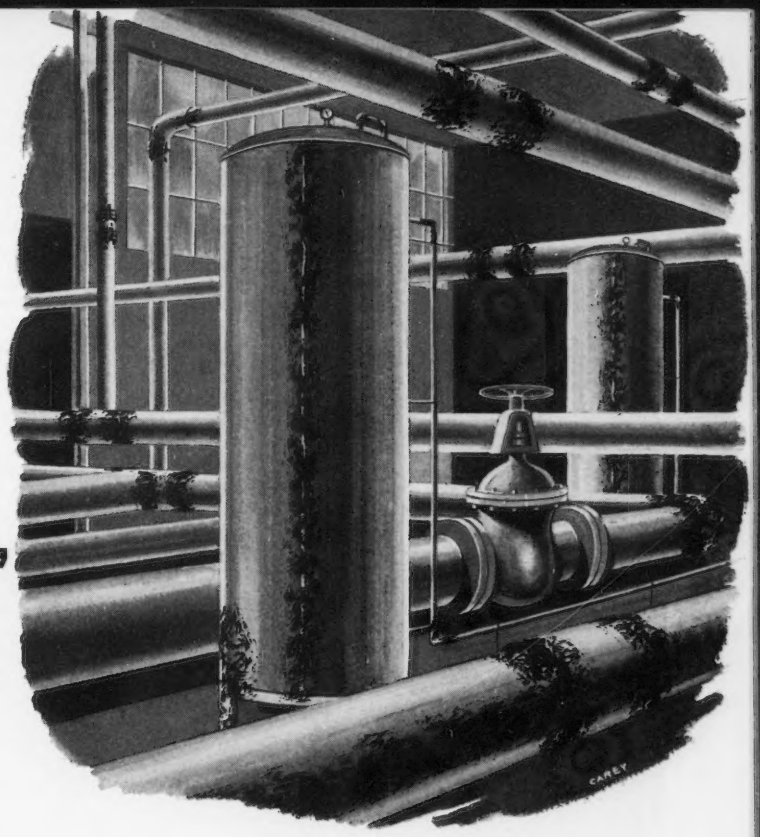

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Growing Interest–

(Continued From Page 112)

steel, now under investigation at North Dakota Agricultural College under Dr. Wouter Bosch is under way. At present pre-rusting of the panels to stages of partial rust (20% of mill scale removed) and complete rust (essentially 100% of mill scale removed) has been completed at the exposure sites at Fargo and Detroit. Panels are now rusting at Kure Beach. It is hoped that paint application can be completed during the spring and summer months.

In the meantime Dr. Bosch is determining moisture vapor permeability and flexibility on these same paints at various stages of aging. This work is being done on free films at film thicknesses approximating those used for panel exposures. It is hoped to correlate free film changes upon aging (both laboratory and exterior) with actual failures on the painted panels.

Plans are under way for the eventual recoating of the panels in the film thickness project. It is thought that only by recoating and re-exposing the panels will an answer be obtained concerning the economics of protection. Thus as panels reach a certain degree of failure they will be retired and stored. Then at some later date all panels will be recoated and re-exposed. After these panels again reach a certain degree of failure a cost of protection per square foot per year can be calculated.

The Montreal Paint and Varnish Production Club has underway a study on the effects of flash rusting on the performances of subsequent coats of paint. Panels were sandblasted, exposed to an industrial atmosphere for various periods of time to allow varying degree of rusting, painted and re-exposed at Sayville, New York. Six months exposures were completed during January, 1957 without any failures. The answer expected from this work is the degree of flash rusting allowable for good paint performance.

The project on film thickness gages as discussed in last year's report to this committee has been concluded. The possibilities for publishing the results were to be discussed at the March 12, 1957 Federation Corrosion Committee meeting.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS—Ed C. Greco, United Gas Corp., Shreveport; G. W. Seagren, Mellon Institute of Industrial Research, Pittsburgh.

A national meeting and five regional meetings were held during 1956. Six short courses were held at Norman, Okla., LaJolla, Calif., Columbus, Ohio; Shreveport, La., Houston, Texas, Los Angeles, Cal.

Publications included the 1952-53 Bibliographic Survey of Corrosion and a 10-Year Index to Corrosion.

Technical committee reports were published on plastics, hydrogen sulfide corrosion, lead cable sheaths, cathodic protection, acids, refinery equipment, anodes, painting specifications and other topics.

There were 39 meetings of technical committees at regional meetings and more than 70 at the national meeting.

New technical committee activities include those on the refinery industry, marine biological deterioration, industrial maintenance paints, coatings equipment, application methods, underground corrosion coordination and numerous others in various corrosion fields.

News stories on the activities of NACE technical committees were published in CORROSION.

NATIONAL BUREAU OF STANDARDS—Delegate: G. A. Ellinger, NBS, Washington.

A very brief summary of the activities of the Corrosion Section for the year 1956 is as follows:

Corrosion Research Council

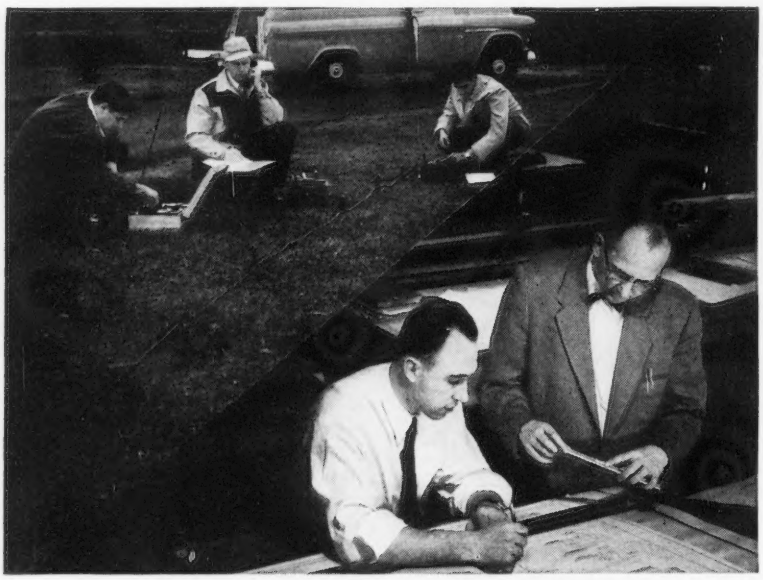
A project "Fundamental Corrosion Reactions at the Surfaces of Metals in Selected Environments" was started during the year.

Mechanism of Corrosion Processes

Several projects involving pitting corrosion, stress corrosion and galvanic corrosion are included under this heading. Two papers were published during the year, both on stress corrosion Relationship Between Crystal Orientation and Stress Corrosion Cracking in Alpha and Beta Brasses by Hugh L. Logan, Research Paper 262, *J. Res. NBS,* **56,** p. 159 (1956) and Stress-Corrosion Cracking of Type 304 Austenitic Stainless Steel by H. L. Logan and R. J. Sherman, Jr., *Welding Journal,* Welding Research Supplement, **35,** p. 389s, 1956. The latter was sponsored by the Pressure Vessel Research Committee of the Welding Research Council. Three other papers are awaiting publication early in 1957.

Other projects on Corrosion of Aircraft Alloys, Corrosion of Steels by Molten Metals and Fly Ash Corrosion of Stainless Steels continued at normal activity rates.

(Continued on Page 116)



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Growing Interest—

(Continued From Page 114)

NATIONAL RESEARCH COUNCIL OF CANADA—Delegate: H. P. Godard, Aluminium Laboratories, Ltd., Kingston, Ont.
The Associate Committee on Corrosion Research and Prevention reports:

Nature of Corrosion Investigation

New samples exposed at the various atmospheric sites in 1956 were: Stelco continuously galvanized steel, Armco aluminized steel Type 2.

A series of steel panels spray metallized with aluminum and zinc has been prepared and will be exposed in 1957.

Panels have been examined after exposures of one and two years of the following metals: Aluminum, (3 alloys); magnesium, (2 alloys); low alloy steel, (3 alloys); stainless steel, (3 alloys). A report on the results is being drafted.

The third annual examination of a series of paint systems on steel was made. A progress report on the results is being drafted. A second series of painted steel panels is being prepared.

Methods for measuring the chloride content of the atmosphere were studied under M. R. Foran of Nova Scotia Technical College. The "wet cloth" method, the "wet candle" method and direct filtering through glass wool were the methods involved. A report on the work was prepared.

Corrosion Research Sponsored at Canadian Universities

In 1956 $9870 was granted by the council to support four corrosion researchers in four Canadian Universities.

Corrosion Meetings

None held in 1956. One planned for 1958.

OFFICE OF NAVAL RESEARCH—Delegate: Julius J. Harwood, Office of Naval Research, Washington.
The following represents the active contract corrosion research program of the Office of Naval Research:

Fundamental Studies of Corrosion—M. G. Fontana, Ohio State University. Studies of passivity of stainless steels and mechanisms of stress corrosion cracking of austenitic stainless steel.

Passivity and Passivation Studies—H. H. Uhlig, Massachusetts Institute of Technology. Studies of passivity of metals and alloys and the relationships between electron configuration, nature of passive films and corrosion behavior of metals.

Adsorption Studies and Corrosion Reactions—N. Hackerman, University of Texas. Studies of adsorption reactions,

(Continued on Page 118)

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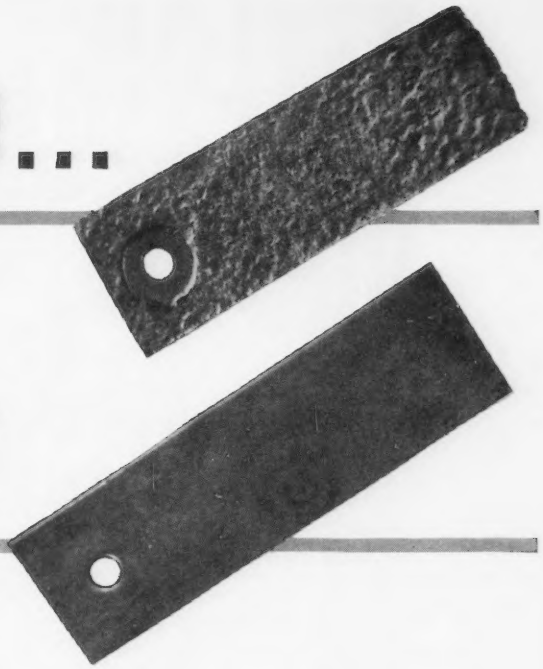

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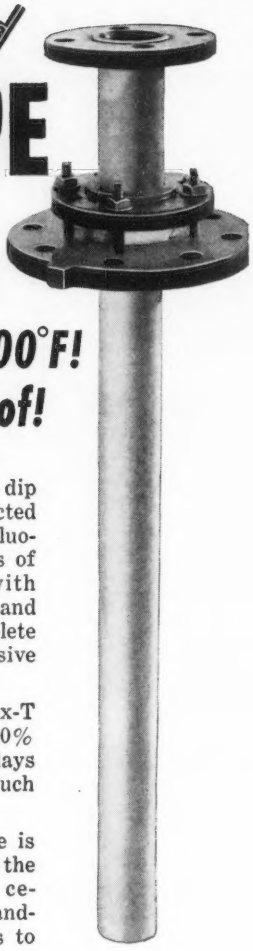

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Growing Interest—

(Continued From Page 116)

polarization behavior, contact potentials and inhibitor action and mechanisms.

Influence of Environment on Failure of Metals—W. D. Robertson, Yale University. Studies of chemical and structural factors associated with accelerated failure of alloys in corrosive environments.

Surface Properties of Single Crystals—A. T. Gwathmey, University of Virginia. Studies of the influence of crystallographic orientation and other factors upon surface reactions.

Corrosion of Iron in Weak Acid Solutions—Crest Research Laboratories, Los Angeles, California. Study of the mechanism of corrosion of iron in weak acid solutions.

Chemical Behavior of Subgrain Boundaries—M. Metzger, University of Illinois. Studies of the effects of solute atoms on the susceptibility of subgrain boundary attack and the influence of sub-grain boundary corrosion susceptibility on stress-corrosion behavior.

Electrochemistry of Hydrogen Evolution and Stress-Corrosion Reactions—J. O'M. Bockris, University of Pennsylvania. Study of the electrochemistry of hydrogen evolution and stress-corrosion processes with emphasis on the factors promoting the adsorption, concentration and evolution of hydrogen on metal surfaces before diffusion and permeation occurs.

Corrosion of Metal Single Crystals and Grain Boundaries of Known Misorientation—H. Paxton, Carnegie Institute of Technology. Studies of the corrosion behavior of single crystals and oriented bicrystals of austenitic stainless steels to elucidate the mechanism of grain boundary corrosion of "sensitized" stainless steels.

"Wash-Primer" Program—E. R. Allen, Rutgers University: Studies of the chemical changes which result in the deterioration of the "Wash-Primer" formulation upon aging. F. R. Eirich, Polytechnical Institute of Brooklyn: Studies of structure, properties, adsorption charcteristics of polymer and the polymer reactions which occur in the formulation. (Both studies are being completed.)

SOCIETY OF NAVAL ARCHITECTS AND MARINE ENGINEERS—Delegate: Paul Ffield, Bethlehem Steel Co., Quincy, Mass.

Two papers on corrosion were presented during the year. One concerning the Navy's cathodic protection activities was given at the annual meeting and the other on propeller deterioriation at New England Section.

The society's Research Panels M-8 on Investigation of Tail Shaft Assemblies and M-11 on Investigation of Propeller Hub Sealing Arrangements have been active. The work of Panel M-11 was summarized in the society's bulletin for July 1956, page 34.

TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY—Delegates: Stephen J. Baisch, Thilmany Pulp & Paper Co., Kaukauna, Wis. and H. O. Teeple, International Nickel Co., Inc., New York.

The Corrosion Committee of the Tappi organization held a meeting to discuss and evaluate the response to a corrosion questionnaire recently sent to member pulp and paper mills. Some 117 re-

(Continued on Page 119)

Growing Interest—

(Continued From Page 118)

turns were tabulated and will form the basis of future corrosion studies by the committee.

At the meeting the following formal papers were presented: Digester Repair to Abate Corrosion by E. W. Hopper and J. B. Morrison. (Published in *Tappi*, 39, No. 11, 793-797 (November, 1956). Corrosion Studies of Carbon Steel in Alkaline Pulping Liquors by the Potential—Time and Polarization Curve Methods, I Theory, Methods, and Selected Results by W. A. Mueller. (Published in *Can. J. Technol*, **34**, No. 3, 162-81, May, 1956).

Eleventh Engineering Conference—Boston, Mass. October 8-11, 1956. The following formal papers were presented: Experiences with Cathodic Protection of Digesters by F. P. Morrison and H. M. Tanavan (to be published in *Tappi*). Electrochemical Corrosion Studies on Carbon Steel in Alkaline Pulping Liquors, II. Mixtures of White with Oxidized or Non-Oxidized Black Liquor by W. A. Mueller (to be published in *Tappi*). Analysis of Corrosion Questionnaire Returns by F. E. Hutton and S. J. Baisch (to be published in *Tappi*).

In committee and open discussion the following items were considered: Second Summary of Alkaline Digester Corrosion by C. W. Smith. It was agreed that at the twelfth Tappi Engineering Conference to be held in Cincinnati, Ohio on September 23-26, 1957 a formal paper will be given outlining the experience and results after three years of regional digester activity. Corrosion Questionnaire—Future Planning by S. J. Baisch. An analysis of the recent corrosion questionnaire indicates, from an economic viewpoint, some 27 corrosion areas existing in the pulp and paper industry. These areas lend themselves into five general categories which form the basis of forming five sub-committees to make further studies and recommendations on the industrial corrosion problems.

Tappi Research Project No. 86 financed jointly by the Institute of Paper Chemistry and Tappi entitled The Corrosive Behavior of Electrolytes in Alkaline Pulping Solutions has made excellent progress. A formal paper on the first part of the work will be presented at the Tappi Engineering Conference in Cincinnati, Ohio on September 23-26, 1957.

U. S. Army Signal Engineering Laboratories, Fort Monmouth, N. J. Representative, H. M. Gade.

Nature of Corrosion Investigations

Galvanic Corrosion. Specimens consisting of magnesium coupled to various dissimilar metals were coated with various finishing systems and exposed to atmospheric conditions in the Northern California coastal area. Observations to date:

a. Galvanic corrosion, where it has occurred on unpainted anodically coated magnesium panels, was localized and resulted in perforations of the magnesium.

b. Paint systems on the anodically coated magnesium portions of the couple were effective in lessening perforations.

c. In many couples the first sign of paint failure occurred on the cathodic portion of the couple rather than on the

(Continued on Page 120)

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TECHNICAL REPORTS

on

Corrosion in Oil and Gas Well Equipment

T-1A Survey of Corrosion Control in California Pumping Wells. A Report of T-1A on Corrosion in Oil and Gas Well Equipment, Los Angeles Area. Pub. 54-7. Per Copy, $.50.

T-1C Current Status of Corrosion Mitigation Knowledge on Sweet Oil Wells. A Report of Technical Unit Committee T-1C on Sweet Oil Well Corrosion. Per Copy, $.50.

T-1C Field Practices for Controling Water Dependent Sweet Oil Well Corrosion. A Report of Technical Unit Committee T-1C on Sweet Oil Well Corrosion, Compiled by Task Group T-1C-1 on Field Practices. Pub. No. 56-3, Per Copy $1.00.

TP-1D Sour Oil Well Corrosion. *Corrosion* August, 1952, issue. NACE members, $.50; Non-members $1 Per Copy.

TP-1G Field Experience With Cracking of High Strength Steel in Sour Gas and Oil Wells. (Included in Symposium on Sulfide Stress Corrosion.) (Pub. 52-3) $1 Per Copy, 5 or more copies to one address, $.50 Per Copy.

T-1G Sulfide Corrosion Cracking of Oil Production Equipment. A Report of Technical Unit Committee T-1G on Sulfide Stress Corrosion Cracking. Pub. 54-5. $.50 Per Copy.

T-1J Status Report of NACE Technical Unit Committee T-1J on Oil Field Structural Plastics. Per Copy $1.00.

T-1J Reports to Technical Unit Committee T-1J on Oilfield Structural Plastics. Part 1, Long-Term Creep of Pipe Extruded from Tenite Butyrate Plastics. Part 2, Structural Behavior of Unplasticized Geon Polyvinyl Chloride. Publication 55-7. Per Copy $.50.

T-1J Summary of Data on Use of Structural Plastic Products in Oil Production. A Status Report of NACE Technical Unit Committee T-1J on Oilfield Structural Plastics. Per Copy $.50.

T-1J Service Reports Given on Oil Field Plastic Pipe. Activities Report of T-1J on Oilfield Structural Plastics. Per Copy $.50.

T-1J Oilfield Structural Plastics Test Data are given. Activities Report of T-1J on Oilfield Structural Plastics. Per Copy $.50.

T-1J Reports to Technical Unit Committee T-1J on Oil Field Structural Plastics. Part 1—The Long Term Strengths of Reinforced Plastics Determined by Creep Strengths. Part 2—Microscopic Examination as a Test Method for Reinforced Plastic Pipe. Per Copy $.50.

T-1J Status Report of NACE Technical Unit Committee T-1J on Oil Field Structural Plastics. Part 1—Laboratory Testing of Glass-Plastic Laminates. Part 2—Service Experience of Glass Reinforced Plastic Tanks. Part 3—High Pressure Field Tests of Glass-Reinforced Plastic Pipe. Per Copy $.50.

T-1K Proposed Standardized Laboratory Procedure for Screening Corrosion Inhibitors for Oil and Gas Wells. A Report of T-1K on Inhibitors for Oil and Gas Wells. Publication 55-2. Per Copy $.50.

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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Hydrogen Sulfide Stress Corrosion Report Issued

Reprints of the report "Hydrogen Absorption, Embrittlement and Fracture of Steel" on the 4-year research program sponsored by NACE Technical Committee T-1G on Sulfide Stress Corrosion Cracking are now available. The 24-page report, replete with tabulated data and liberally illustrated, was written by W. D. Robertson, Hammond Metallurgical Laboratory, Yale University and Arnold E. Schuetz, Research Dept., Bridgeport Brass Co., Bridgeport, Conn.

The report was published in CORROSION, July, 1957 as NACE Publication 57-17. Copies are available at $2, remittance in advance, from National Association of Corrosion Engineers, 1061 M & M Bldg., Houston 2, Texas.

Contributors of funds to carry out the research were:

The Atlantic Refining Co., Axelson Manufacturing Co., Bethlehem Steel Co., California Research Corp., Cameron Iron Works Inc., Cities Service Research and Development Co.

E. I. DuPont de Nemours and Company, Inc., Gulf Oil Corp., Humble Oil and Refining Co., The International Nickel Company, Inc., Magnolia Petroleum Co., Phillips Petroleum Co., Pure Oil Co., Shell Development Co., Sinclair Research Laboratories, Inc.

Standard Oil Development Co., Sunray Oil Corp., Tidewater Oil Co., Union Oil Company of Calif., Union Producing Co.

Corrosion Was Problem In Test of Solar Heating

Corrosion was one of the problems encountered in the tests of a solar heating system for an office building at Alburquerque, N. Mex. F. H. Bridgers, D. D. Paxton and Roger W. Haines, reporting on the system installed in their own offices last year at an ASME meeting June 12 in San Francisco said it performed satisfactorily through the worst part of the winter, including a much cloudier than normal January.

Growing Interest—

(Continued From Page 119)

magnesium, indicating that additional effort should be spent in protecting the cathodic member of the couple.

d. Couples with alkyd enamel finish appear to fail sooner than couples with phenolic or epoxy enamel.

Tropical Field Service Life of Selected Electronic Components

This investigation was initiated by the U. S. Army Signal Corps to obtain data on the behavior of various electronic components and materials under simulated and natural tropical exposure. The ultimate objective is to use the data for establishing laboratory test procedures for predicting the reliable tropical service life of the components. Field exposures were made at three different locations in Panama; Beach, open jungle, and shady jungle, representing the range of environmental conditions encountered in the tropics. Nineteen types of military electronic components and materials in the categories of resistors, capacitors, r.f. chokes, i.f. transformers, rectifiers, and terminal boards are being investigated.

See August CORROSION for reports from Foreign Correspondents of the Inter Society Corrosion Committee on control work being done in Australia, Belgium, Denmark, France, Germany, Italy, Japan, New Zealand, Spain, Switzerland and the United Kingdom.

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GENERAL NEWS

Japanese Plan to Form Corrosion Committee

Efforts are being made to organize in Japan a group similar in function to the Inter Society Corrosion Committee of the National Association of Corrosion Engineers. Dr. T. Fujiwara, Imperial Chemical Industries (Japan) Ltd., Tokyo reported to the ISCC that representatives of some of the principal technical organizations, educational institutions and industrial organizations have indicated interest in forming such an organization.

The ISCC consists of delegates from 14 engineering, trade organizations and technical groups. A principal function of the committee is to facilitate cooperation among affiliated organizations to improve dissemination of information and prevent redundant work.

Auto Exhaust, Incinerators Most Pressing Smog Items

Control of automobile and incinerator gases was given as "The most important goal in the smog fight" by the Air Pollution in its Third Technical Progress Report. Copies of the report are available from the foundation office, 704 South Spring St., Los Angeles 14 at $3 a copy.

$4000 Allocation Made to Corrosion Research Council

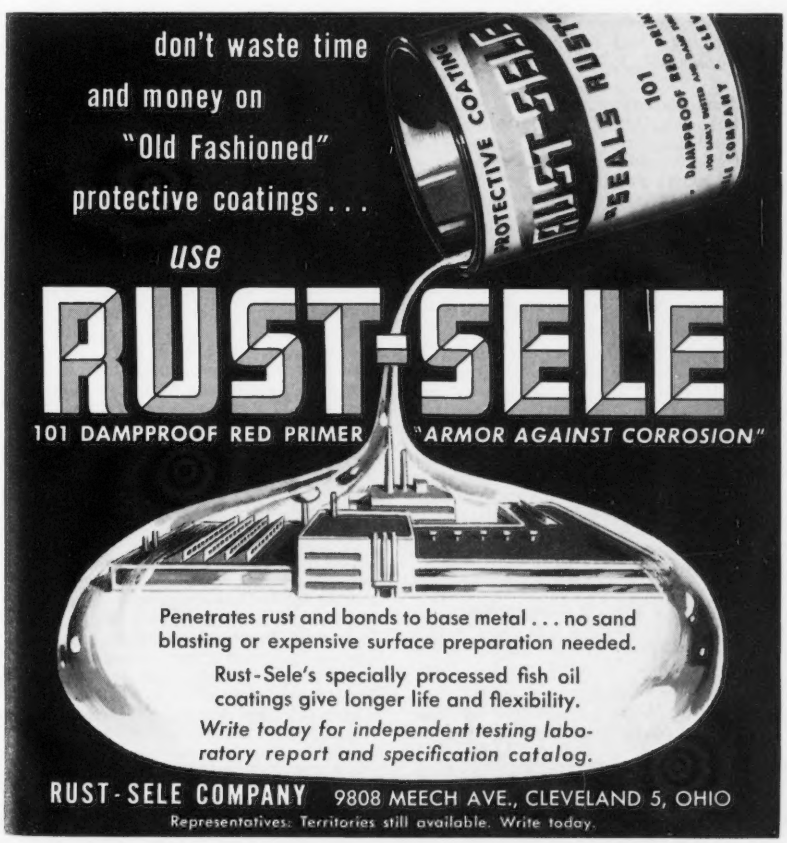
Allocation of $4000 to the Corrosion Research Council, a project sponsored by the American Institute of Mining, Metallurgical and Petroleum Engineers has been made by the United Engineering Trustees Engineering Foundation. The sum is intended to further investigation started last year into fundamental corrosion reactions.

The Corrosion Research Council and National Bureau of Standards, cooperating in the fundamental research, also have been engaged in a second joint project, exploration of the relation of stress to the corrosion of metals, which includes a study of the role of hydrogen.

Another AIMPE project, "Alloys of Iron Research, will receive $5000. It is publishing in June a fourth monograph in the series "Boron, Calcium, Columbium, Tantalum and Zirconium in Iron and Steel."

Standards Conference Set By ASA November 13-15

The Eighth National Conference on Standards will be held November 13-15 concurrently with the 39th Annual Meeting of the American Standards Association at San Francisco.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Field Tests Urged in Final Selection of Industrial Coatings

Field tests were recommended for the final selection of industrial paints after initial laboratory screening by William C. Naumann, Shell Chemical Company at the April 26-27 Southwestern Paint Convention in Houston. Mr. Naumann's talk "A Modern Approach to Corrosion Prevention through Protective Coatings," covered surface preparation, systems, application methods and materials selection—the latter in detail.

Naumann

Technical activities included a panel discussion presented by the Pigments Committee of the American Zinc Institute composed of J. A. Reising, St. Joseph Lead Co., moderator; A. C. Elm, New Jersey Zinc Co.; Paul Whitford, Eagle-Picher Company and Clovis H. Adams, Sherwin-Williams Company.

Numerous other discussions were held including talks by officers of the Federation of Paint and Varnish Production Clubs and the National Paint, Varnish and Lacquer Association.

BOOK NEWS

Symposium on Corrosion Fundamentals. Edited by Anton de S. Brasunas. 255 pages, 6 x 9¼ inches, cloth. 1956. University of Tennessee Press, University of Tennesse, Knoxville. Per copy, $5.

Collected papers presented March 1-3, 1955 at the University of Tennessee at a program sponsored by the University and Southeastern Region, National Association of Corrosion Engineers. The focus is on fundamentals.

Papers included are: Corrosion and Metal Structures, E. E. Stansbury; Nature of Corrosion, A. de S. Brasunas; Atmospheric Corrosion, H. R. Copson; Coatings for Atmospheric Corrosion Protection, J. I. Richardson; High Temperature Corrosion, Henry Inouye;

Liquid Metal Corrosion, E. E. Hoffman; Principles of Cathodic Protection, Milton Stern; Corrosion Problems in Steam Power and Industrial Boiler Plants, R. M. Lemen; Corrosion Failures in Chemical Plants, Mars G. Fontana; Inhibition of Metallic Corrosion in Aqueous Media, Harry C. Gatos; Passivation of Stainless Steel, N. A. Nielsen;

Influence of Mechanical Factors on Corrosion, J. J. Harwood; Geometric Factors in Electrical Measurements Relating to Corrosion and Its Prevention, W. J. Schwerdtfeger and I. A. Denison; Cathodic Protection Principles and System Design, H. C. VanNouhuvs; Use of Plastics and Plastic Liners for Corrosion Protection, R. B. Seymour.

Equipment Services NEW PRODUCTS Materials Literature

Haveg Industries, Inc. has developed a method of fabricating asbestos filled phenolic or furan, graphite filled epoxy and glass reinforced polyester tanks inside existing tanks. The existing tank can be retained as support, or removed in some instances. The company also uses pre-molded plastic sections which are assembled and cemented together inside a plant.

•

Randolph Company, 1018 Rosine St., Houston has developed a pump involving the use of a flexible tube through which corrosive liquids may be pushed by squeegee action of pump parts. No part of the pump contacts the corrosive.

•

Fabri-Valve Company of America, Portland, Oregon is ready to mass produce 6-inch wedge gate titanium valves, principally for the pulp and paper industry. The valve, weighing about 60 pounds, is 40 pounds lighter than a comparable steel valve.

•

Titanium and Zirconium ingots and billets up to 2400 pounds for titanium and 2800 pounds for zirconium are available from Oregon Metallurgical Corp., Albany, Oregon. They are available alloyed with chromium, manganese, tantalum, columbium, platinum or other metals.

•

Protective Coatings Division, Pittsburgh Coke & Chemical Company has developed a device which can deposit a non-sagging coating of Tarset up to 20 mils thick in a single pass inside pipe. The applicator consists of a spinning head into which the coating and catalyst are introduced just before application, mixed thoroughly and thrown out by centrifugal force onto pipe interiors. The device, available to interested companies from PCC can coat the inside of 20-foot lengths of 3, 4 and 6-inch pipe.

•

Nialite, a zinc-free manganese bronze was used to fabricate a 21 foot, 8-inch propeller weighing 49,300 pounds for the S. S. President Adams, a Marine class vessel of American President lines. The new wheel, lighter and stronger than those made with other materials, is expected to last longer and to give substantial fuel savings because of improved efficiency resulting from thinner blades. Resistance to cavitation of 6 to 1 over manganese bronze is expected.

•

Abrasive Shot & Grit Co., Springville, N. Y. has published a 16-page manual covering SAE specifications of abrasive shot and grit. Cleaning methods are illustrated.

•

Mannesmann Tube Co., Ltd., a $20 million, highly mechanized mill for production of seamless steel tubing has been opened for production at Sault St. Marie, Ont. A labor force of 500 men is expected to produce 225,000 tons of finished seamless steel tubes annually. The plant is one of 70 plants controlled by the Mannesmann group.

Ebasco Services Inc. has opened an office at 120 Montgomery St., San Francisco as headquarters for a staff of consultants on the West Coast.

•

Production Tests in the use of chemical coatings for the prevention of corrosion can be made at a new "production line" laboratory opened by Parker Rust Proof Co. of Detroit at a cost of $300,000.

•

Twin-Tuffy, a portable spray painting unit which delivers 4.6 to 5 cubic feet of air per minute, weighs 59 pounds and was designed by deVilbiss Co., Toledo, Ohio as a high performance unit for professional jobs at low cost.

•

Oxygen, acetylene and nitrogen will be produced at a new National Cylinder Gas Co. plant at Jackson, Miss. adjacent to the mill of the Mississippi Steel Corp.

•

Sulfonate OS, a sodium salt of the oil-soluble fractions of sulfonic acids, which is miscible in petroleum oils and colloidally dispersable in water is recommended for the manufacture of oil-soluble rust and corrosion inhibitor preparations. The anionic surface active agent is a product of Sun Oil Company's Industrial Products Dept., 1608 Walnut St., Philadelphia 3, Pa.

•

Stevens Institute of Technology has started construction of a $2 million seven story science and engineering building at Hoboken, N. J.

•

Bradford Laboratories, Bradford, Pa. has been purchased by Hagan Chemicals and Controls, Inc. to broaden its penetration into water conditioning to include water flooding.

•

Alcoa has selected a 2000-acre site in Westmoreland County, 28 miles east of Pittsburgh as the site for a new research and development center.

•

Gates Engineering Company's outstanding capital stock has been exchanged for stock of Macco Chemical Co., Cleveland. The Gates firm will continue to operate as a separate corporation.

•

Flexible Polyethylene Plastic Pipe intended for use with portable water may now be classified under a new commercial standard of the U. S. Department of Commerce. CS 197-57, effective April 15 requires that pipes meet specifications of National Sanitation Foundation. Polyethylene Pipe Standards Committee of the Society of the Plastics Industry cooperated in producing the new standard. A schedule of recommended working pressures is included.

•

Fused-Film Wire, produced by Inso Products, Ltd., Union, New Jersey, has a 0.002-inch film of Teflon which makes it unusually resistant to water, chemicals and fungus. The wire is being produced principally for use in electrical and electronic equipment.

MEN in the NEWS

Fred L. Plummer, Director of Engineering, Hammond Iron Works, Warren, Pa. was awarded the Samuel Wylie Miller Memorial Award of the American Welding Society at its April meeting in Philadelphia. **Dewitt C. Smith,** Chief Metallurgist, Harnischfeger Corp., Milwaukee, gave the Adams Lecture and honorary membership in the society was given to **O. B. J. Fraser,** Assistant Manager, Development and Research Div., International Nickel Co., Inc., New York.

•

Edward J. Dunn has been appointed chief project chemist in the Technical Department at American Viscose Corporation's Marcus Hook cellophane plant.

•

Gerald L. Lee has transferred to the Iron and Non-Ferrous Castings Section of the International Nickel Company's Development and Research Division in New York. He spent 12 years at Inco's Bayonne laboratory.

•

James S. Anderson, formerly general sales manager of the Tubular Products Division, The Babcock & Wilcox Company has been elected a vice-president of the company in charge of sales for that division.

•

Dudley D. Fuller, professor of mechanical engineering at Columbia University and Principal Scientist at the Franklin Institute of Pennsylvania has been given the American Society of Lubrication Engineers' National Award, which constitutes life membership in the society. **Douglas Godfrey,** California Research Institute was awarded the Capt. Alfred E. Hunt Memorial Medal for the best paper delivered before the society by a member or published in the society's journal during the year. **Ernest Rabinowicz,** Massachusetts Institute of Technology received the Walter D. Hodson certificate and cash award for the best paper presented by an ASLE member 30 years of age or less during the year.

•

George H. Williamson has been elected a vice-president of American Chemical Paint Co. in charge of West Coast activities.

•

Clifford F. Rassweiler, vice-chairman of the board and vice-president for Research and Development of Johns-Manville Corp. has been given the 1957 Industrial Research Institute medal for his work in two great corporations and stimulation of industrial research. Dr. Rassweiler was with DuPont when alkyd vehicles were developed as substitutes for oleoresinous vehicles for paints.

•

William M. Haile has been appointed president of Linde Company, a Division of Union Carbide Corp.

TECHNICAL TOPICS

A Liquid Additive to Limit Oil Ash Corrosion*

Abstract

The problem of vanadium and sodium corrosion of fire side of boilers and of gas turbines and Diesel engines is reviewed briefly and some explanation of the growing severity of the problem given. Elements of the theory whereby reduction of corrosion by increasing the melting temperature of oil ash residue are explained, and some of the materials used for this purpose named. Advantages and efficiency of using an oil soluble aluminum salt inhibitor over inhibitors introduced as slurries are described and some experimental results tabulated.

MAJOR PROBLEMS created when Bunker "C" or No. 6 fuels are burned in Diesel engines, gas turbines or under boilers are corrosion and deposits which result from combustion. These residual fuels are the residue remaining after the refineries have processed crude oil to remove gasoline, kerosene, distillate fuels and lubricating oils. It is necessary either to heat the residuum or to blend it with a cutter stock in order to pump it to storage.

Figure 1 shows refinery production of residual fuels in the United States, together with U. S. demand and imports and exports over the past decade. It is interesting to note that, although total runs of crude oil to refineries have increased enormously to supply the demands for gasoline and distillate heating oils and Diesel fuels, U. S. production of residual fuels has not been sufficient to meet demand, thereby necessitating imports.

Harmful Compounds Present

In many cases imported residual fuels and foreign crude oils refined in the United States contain considerable quantities of sulfur, vanadium and sodium. These components have caused frequent and severe corrosion and harmful deposits in Diesel engines, gas turbines and boilers. Table 1 shows the vanadium and sodium content of some typical fuels from U. S. refineries processing South American crudes, as well as their content in other refineries.

In most cases fuels in Table 1 are not full-viscosity No. 6 fuels, but rather are of intermediate viscosity, blended in the range of 250 to 650 SSU at 100 F. They are representative of the fuels bunkered by a number of large motor ships operating from U. S. East and West coast ports in world-wide trade.

Full-viscosity No. 6 fuels, from which those of intermediate viscosity are blended, contain considerable more vanadium and sodium because dilution of these metals is in proportion to the amount of cutter stock used.

Table 2 shows the metal content of some typical No. 6 fuels.

Figure 2 shows how vanadium content of No. 6 fuel oils received during the past ten years by a large electric public utility for use in oil-fired boilers gradually increased.

Central Station Corrosion Problems

Operators of central stations are experiencing more corrosion of superheater spacers and hangers and deposits on tubes and other surfaces which cannot be removed by soot blowers. Figure 3 is a photograph of a tube section, showing metal wastage and reduction in wall thickness which caused it to rupture in service. This wastage does not occur generally except in high temperature boilers where the metal skin temperature approaches 1200 F. Fireside temperature of tubes normally would not be more than 50 F to 75 F higher than that of steam passing through the tubes, but spacers, hangers and baffles are not cooled by these vapors and therefore are exposed to combustion products at prevailing gas temperatures.

TABLE 1—Vanadium and Sodium Content of Some Typical Blended Residual Type Fuels

Sample	Source	Parts per Million	
		Vanadium	Sodium
A	U.S. Refinery[1]	200	250
B	U.S. Refinery[1]	250	40
C	U.S. Refinery[1]	300	100
D	U.S. Refinery[1]	150	100
E	U.S. Refinery[1]	200	200
F	U.S. Refinery[2]	90	80
G	U.S. Refinery[3]	30	70
H	U.S. Refinery[1]	200	10
I	Middle East	60	10
J	Far East	20	60
K	Far East	150	90

[1] South American crude.
[2] United States crude.
[3] United States (California) crude.

Conditioning and Treatment

It appears that one effective approach to the problem of corrosion by vanadium and sodium is to remove as much of the sodium as is feasible economically by water washing and to inhibit the remainder with magnesium, calcium, barium, or aluminum salts or oxides. A few of the metallic inhibitors which have been tried with varying success include magnesium sulfate, magnesium oxide, calcium oxide, barium acetate, aluminum oxide, dolomite and kaolin. Relative effectiveness of these materials, their cost, method of introduction and the quantities required are not conclusively established.

Cost of materials and the capital expenditure for mixing and injection equipment must be balanced against the replacement and outage costs attributed to the corrosion and deposits due to the vanadium, sodium and sulfur in the fuel. To convert a gas turbine operating on distillate Diesel fuel to No. 6 fuel might effect a fuel savings of 4 cents per gallon, in which case a treatment cost of 1 cent per gallon might be justified. On the other hand, however, a central station operating boilers on No. 6 fuel might find it difficult to justify a fuel

(Continued on Page 124)

TABLE 2—Vanadium and Sodium Content of Some Typical No. 6 Fuels

FUEL	PARTS PER MILLION	
	Vanadium	Sodium
L	90	200
M	150	40
N	500	90
O	224	37
P	800	60

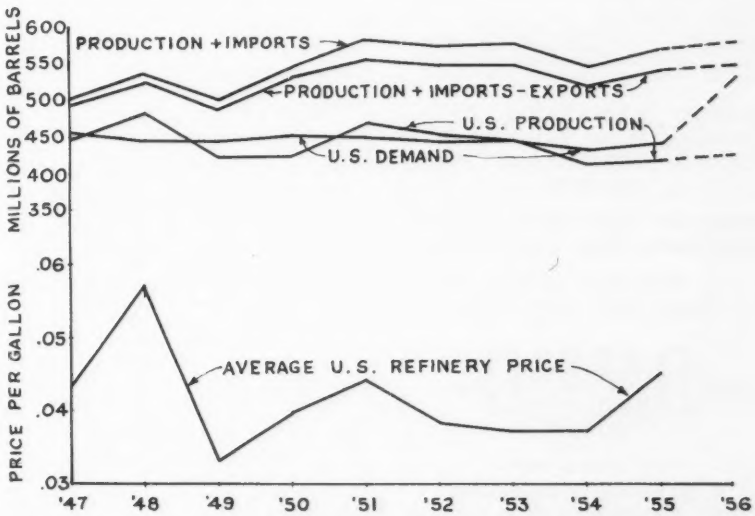


Figure 1—Supply, demand and price of residual fuel oils in the United States for 10 years.

* A paper presented by R. S. Norris at a meeting of T-5B-3 on Oil Ash Corrosion at St. Louis, Mo. during the NACE 13th Annual Conference March 11-15, 1957 under the title "A New Additive Approach to the Oil Ash Corrosion Problem."

A Liquid Additive–

(Continued From Page 123)

treatment cost of 0.1 cent (1 mill) per gallon. In the case of Diesel engines converted to residual fuels or blends, savings in fuel costs of as much as 40 percent may be effected so an additive cost of 0.2 cents (2 mills) may be justified to minimize corrosion and wear.

Damage to Diesels Serious

In the Diesel engine using fuel sample "C," a considerable deposit was observed on the piston heads and spectrographic analysis showed it to consist of the following: Vanadium, 26.07%; sodium, 2.13%. Because destructive tendencies of the vanadium and sodium components of this fuel were inhibited by an additive, no corrosion of the piston head was indicated.

Figure 4 shows the type of attack which can be caused by the vanadium and sodium in residual fuel oils when metal temperatures are in the range where oil-ash corrosion occurs. Cases have been observed where metal wastage

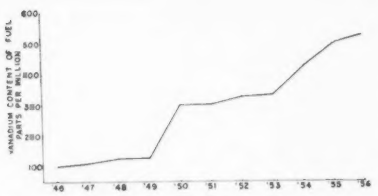
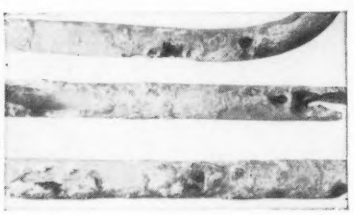


Figure 2—Vanadium content of fuels received by a public utility during the past decade.

CORROSION PROBLEMS?



SULFUR, VANADIUM, AND SODIUM, FROM THE FUEL OIL CAUSED THESE BOILER TUBE FAILURES

BARSAD FUEL TREATMENT PREVENTS THIS CORROSION

THIS MAY BE THE TREATMENT YOU NEED

BARSAD

T.M. Reg. U.S. Pat. Off.
Pat. Pending

FOR DETAILS WRITE

R. S. Norris & Associates
LARCHMONT, N. Y.

Figure 3—Boiler tube section, showing metal wastage and reduction in wall thickness.

as deep as 7/8-inch into the piston head has occurred. Areas of maximum metal wastage are not necessarily in the direct path of the injector spray pattern.

Authors of recent papers have shown photographs of Diesel engine exhaust valve corrosion attributed to fuel ash. Fuel used in one of these engines was a blend containing 45 ppm vanadium.

Figure 5 shows a severe case of exhaust valve corrosion and guttering, caused first by the oil-ash corrosion of the valve seat and face followed by a deposit build-up which prevented the valve from seating and a resultant valve blow. Fuel used in this Diesel engine was a No. 4 oil containing 30 ppm vanadium and 90 ppm sodium.

Methods of Applying Inhibitor

To effectively use the inhibitive effects of the metallic or inorganic materials commonly added to fuels to minimize the bad effects of oil-ash corrosion, the slurries or powders must be brought into intimate contact with the oil. If oxides or finely ground powders passing a 325 mesh screen are used, each particle of material is roughly 44 microns in size. Often this does not permit their coming into contact with a sufficiently high percentage of the vanadium and sodium components in the oil so their inhibitive effect is limited.

But if the additives are available in liquid form, with a maximum particle size of 2 microns, it is obvious that more intimate contact between the additive and the fuel is possible. Laboratory fusion tests, for example, have shown that magnesium oxide introduced as a slurry into the fuel at a Mg/Na ratio on the order of 6 magnesium to 1 sodium is less effective in increasing ash fusion temperature than an oil soluble aluminum salt inhibitor in a mol ratio of Al/Na of 0.75/1. Table 3 shows the effect of the two methods of applying the additive on the ash fusion temperature of a high sodium (417 ppm) content fuel and a high vanadium (375 ppm) content fuel.

Minimum capital outlay is required

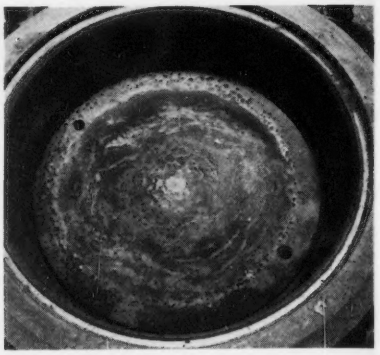


Figure 4—Diesel engine piston head showing corrosive attack attributed to vanadium and sodium in the residual fuels burned.

Figure 5—A severe case of Diesel engine exhaust valve corrosion attributed to fuel ash.

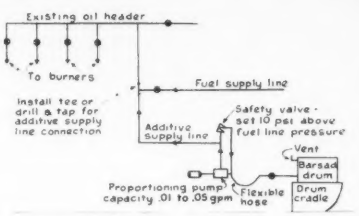

Existing oil header
To burners
Fuel supply line
Install tee or drill & tap for additive supply line connection
Additive supply line
Safety valve - set 10 psi above fuel line pressure
Vent
Barsad drum
Proportioning pump capacity .01 to .05 gpm
Flexible hose
Drum cradle

Figure 6—Schematic of method of introduction of oil-soluble additive into existing fuel oil piping.

TABLE 3—Effect on Ash Fusion Temperature of Additions of MgO and Aluminum Salt Inhibitor*

MATERIAL	Melting Point Degrees F
High Sodium Fuel A	1720
Fuel A + MgO (Mg/Na = 2)	1715
Fuel A + Aluminum Salt Inhibitor* (Al/Na = 0.75)	> 2250
High Vanadium Fuel B	1225
Fuel B + Aluminum Salt Inhibitor* (Al/V = 1.92)	> 2400

* Barsad, R. S. Norris & Associates.

for equipment to introduce a liquid additive into fuel. For example, it may be proportioned into fuel as delivered into storage. It also may be injected into a fuel line serving one unit, such as a particularly troublesome boiler or a gas

(Continued on Page 126)

There's a CORBAN to Combat CORROSION in *Any* Well

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Ceramic Coatings Reduce Creep Rate of Some Alloys as Much as 50%, NBS Reports*

REFRACTORY-type ceramic coatings have come into widespread use on metal parts exposed to high temperatures, as in aircraft engines. The coatings protect the metal at high-temperatures against the various corrosive atmospheres in which these parts normally operate and also affect the creep rates of some alloys under stress at high temperatures. The National Bureau of Standards recently undertook a study of this creep behavior under different temperature-stress conditions with results indicating that under some conditions a coating can reduce the creep rate as much as 50 percent, while under other conditions, a deleterious effect is observed. The investigation was carried out for the Wright Air Development Center by J. R. Cuthill, J. C. Richmond, and N. J. Tighe of the Bureau's enameled metals laboratory.

Creep characteristics of a metal can limit its useful life; thus the effect of ceramic coatings on creep properties is of considerable practical importance. NBS ceramic coating N-143, which contains cerium oxide for refractoriness, chosen for this investigation, was applied to specimens machined from thin sheet metal. Alloys studied are representative of materials that have good high-temperature oxidation resistance including two 80Ni-20Cr alloys (designated as "Lot A" and "Lot C") differing significantly in manganese, iron and silicon content. Both coated and uncoated samples were tested in a conventional controlled-temperature furnace designed for creep testing. Extension of the specimens was measured by sighting with a telescope through a window in the furnace onto a platinum extensometer fastened along the gage length of the specimen.

Of the various combinations of stress and temperature imposed, those at 1975 F and 600 and 1200 psi showed the greatest reduction in creep rates of the ceramic-coated specimens when compared with the uncoated specimens. Under these conditions the beneficial effect of the coating on the creep behavior of both 80 Ni-20 Cr alloys is sufficient to be of practical importance. At 1975 F and 600 psi the creep rate of uncoated Lot A specimens is higher than that of Lot C. However, the reduction in creep rate imparted by the ceramic coating is so much greater in the case of the Lot A alloy that the creep rate of the coated Lot A specimens is significantly lower than that of the coated Lot C specimens.

Void Formation Observed

Extensive internal void formation was observed in the uncoated specimens tested at 1975 F and at 1900 F. Analysis showed that the uncoated specimens lost twice as much chromium as the coated specimens that were tested at the same temperature. Very little void formation was observed in the coated specimens which had been tested at either of these temperatures. Furthermore, no void formation was observed in coated or uncoated specimens tested at 1800 F.

At 1900 F and 900 psi and at 1975 F and 600 psi the creep rate was virtually constant for the entire duration of the test for both Lot A and Lot C alloys. However, at 1800 F and 2200 psi the initial creep rate was comparatively low. After about 20 to 30 hours the creep rate rapidly increased for a few hours and then became gradually less again.

At 1800 F and 2200 psi, the initial creep rate of ceramic-coated specimens was well below that of the uncoated specimens. The creep curves of the coated specimens usually remained below the curves of the uncoated specimens during the first 20 hours. However, during a period of rapidly increasing creep rate beginning about 20 to 30 hours after the start of the test, coated specimens generally showed an even greater rate of increase than the uncoated; fracture of the coated specimens usually occurred during this period. One ceramic-coated specimen, which did not fail under test for more than 1200 hours, showed significantly greater creep after the first 100 hours than did the uncoated specimens.

Coating Insufficiently Fluid

In general, the results suggest that the N-143 coating is not sufficiently fluid at 1800 F to follow the deformation of the specimen at the very high strain rates occurring in these alloys when a stress of 2200 psi is applied and that it therefore cracks. Profuse cracking of the coating was observed in all of the coated specimens that failed in test at 1800 F and 2200 psi but in no other specimens. Presumably, at higher temperatures the N-143 coating, being more plastic than at lower temperatures, was able to follow the deformation of the specimens at strain rates as high as those observed at 1800F and 2200 psi.

TABLE 1—Report of Chemical Analyses[1] of the Two 80 Ni-20 Cr Alloys

Constituent	WEIGHT PERCENT 80Ni-20Cr (Lot A)	80Ni-20Cr (Lot C)
Cr	19.14	19.67
Mn	1.80	.24
Si	1.28	.82
Fe	.61	.10
Co	[2]	.07
Al	.10	.01
Zr	.09	.050
Ca	.038	.032
Cu	.03	.04
C	.03	.02
P	*	.01
Pb	*	.002
S	*	.001
B	*	.0005
Ni	bal.	79.04

[1] Furnished by manufacturers of the respective alloys.
[2] Not reported.

TABLE 2—Composition of the NBS No. N-143 High-Temperature Ceramic Coating

Mill batch composition of the N-143 Coating:

Ingredient	Parts by Weight
NBS Frit No. 435	67½
Cerium oxide, CeO_2	32½
Enameler's Clay	5

Computed melted oxide composition of Frit No. 435:

OXIDE	Mole Percent
SiO_2	51.02
BaO	25.51
BeO	10.20
P_2O_5	2.04
CaO	6.12
ZnO	5.10

A Liquid Additive—

(Continued From Page 124)

turbine operating at high temperature.

Figure 6 shows necessary equipment to be a proportioning variable stroke pump, plus necessary piping and valves.

A liquid, oil-soluble additive does not increase the ash burden of a boiler or gas turbine to the extent that a metal salt or oxide introduced as a slurry or powder often does. A rule of thumb for the introduction of a magnesium salt into fuel calls for a quantity such that the ratio of magnesium weight to vanadium weight in fuel ash should be not less than 3 to 1. To accomplish this, an aqueous solution of magnesium sulfate is injected into the fuel ahead of combustion. Because water droplets containing the additive are suspended in the oil, it follows that an unfavorable particle size ratio exists which limits intimate contact between the magnesium and vanadium.

Fight Fire With Fire

If the best solution to oil ash corrosion is to raise the melting point of the ash by adding high melting point inorganic compounds, it is logical to assume that success may rest as much on the intimacy of contact between the additives and the corrosives in the oil as on the type of metal used. A liquid, oil-soluble, easily applied additive seems to have the ability to disperse quickly into the fuel and to give better contact with vanadium and sodium corrosives than is achieved when additives are introduced as slurries or powders.

* For further technical information, see The Effect of a Ceramic Coating on the Creep Behavior of Some High-Temperature Alloys, by J. R. Cuthill, J. C. Richmond and N. J. Tighe, *J. American Ceramic Society* (in press).

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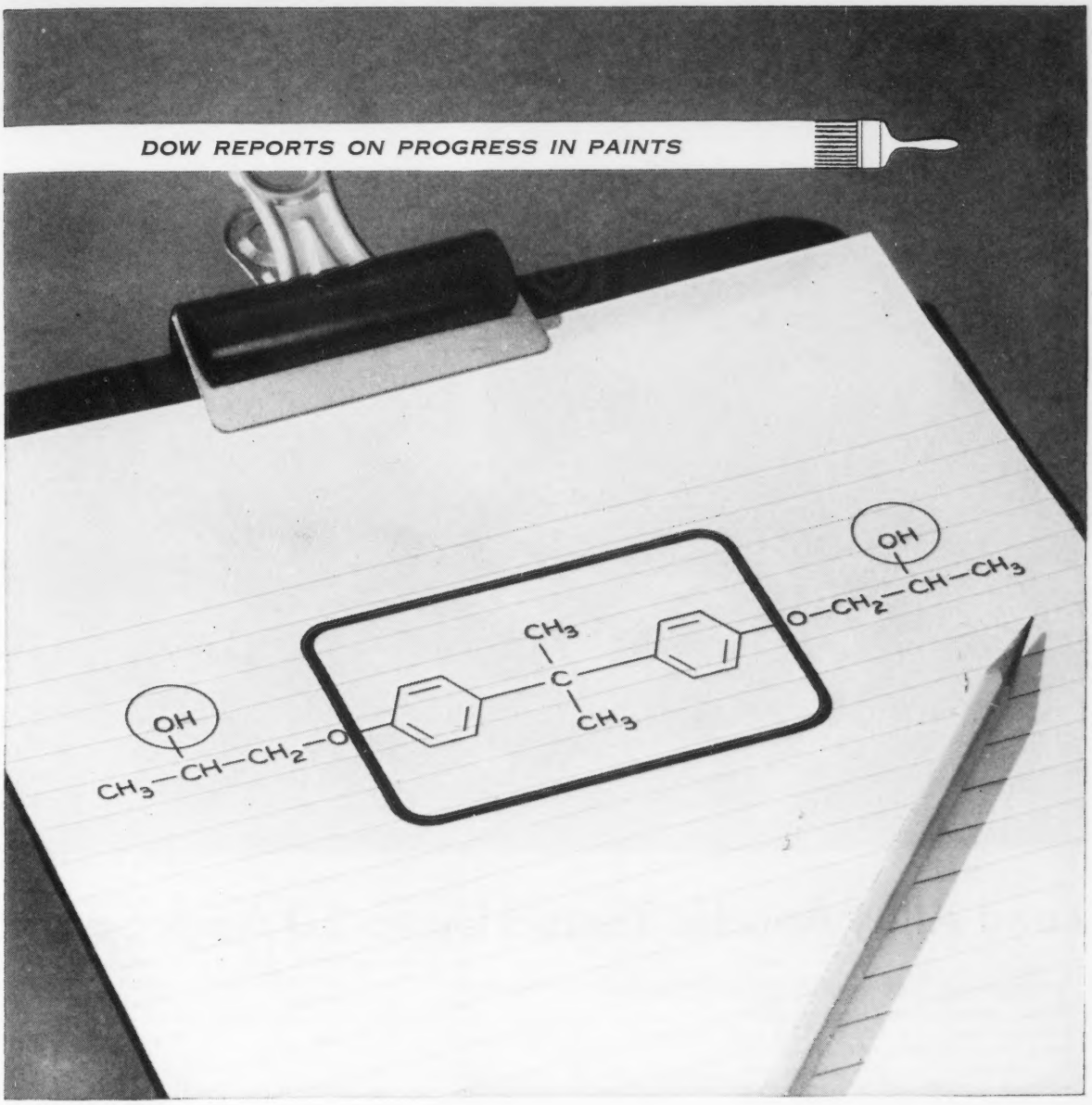

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Lead Alloy Anodes Tested Up to 20 Amp. Sq. Ft.*

ALTHOUGH the most viciously corrosive bulk electrolyte is sea water cathodic protection has been successful in it for more than a century. This has been achieved using sacrificial anodes of zinc, magnesium or aluminum and inert anodes of carbon and platinum. Economic considerations show that it often is advantageous to use impressed current for this work, particularly where either space or other engineering considerations made the mounting and replacement of sacrificial anodes difficult.

In mid-1954 the author and his colleagues began to investigate suitable materials for inert anodes in sea water. Graphite, scrap iron, silicon-iron and platinum were tried but none proved ideal. It seemed that a lead alloy could be used successfully if a conducting peroxide film could be formed on its surface.

Research began late that year with an 8 percent antimonial lead alloy. The peroxide film was formed at current densities above 20 amperes per square foot in artificial sea water and the metal consumption was low, being approximately 2 lb. per ampere year. Below this current density the brown peroxide did not form and the metal was rapidly consumed.

Contemporarily with this work the British Admiralty had begun to investigate another series of lead alloys and both parties felt that some discussion and exchange of results would be mutually beneficial. Following these talks research continued and an alloy of exceptional properties was evolved which has been termed "CX3" alloy.

CX3 alloy retains most of the properties associated with strengthened lead, being a good conductor, malleable, and electrical connections can be made to it readily. It can be extruded and cast with or without inserts or can be cold worked by swaging or other techniques. The alloy is readily machined and pieces may be fused together by ordinary jointer's methods.

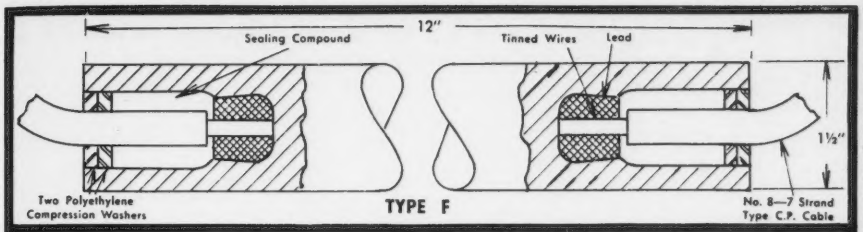
Tests have been carried out both in the laboratory and in the field on some hundred test runs. Anodes used for this purpose were cast in the laboratory by pouring molten lead into standard Pyrex test tubes. The anodes weighed about 100-150 grammes and a self-tapping screw was used as a connector, this being sealed with PVC tape and paraffin wax. Laboratory tests have been made in sea water, diluted sea water and fresh water at a variety of temperatures and in sand and mud. Current densities employed in these tests have varied from 1 to 20 amperes per square foot.

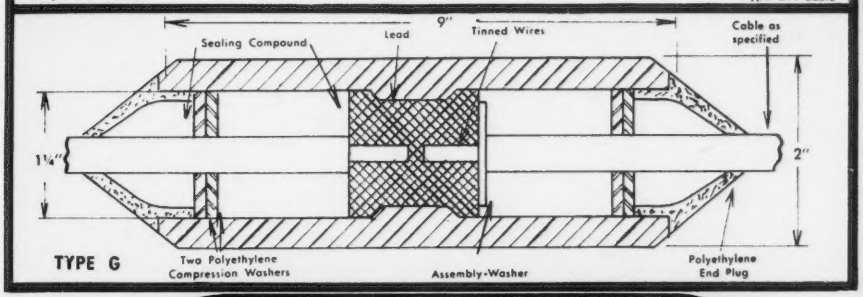
Sea Water Tests. A CX3 alloy anode begins by emitting large volumes of a white corrosion product and, after 3-4 ampere hours per square foot, a brown peroxide layer forms. Loss of metal, at first great, decreases rapidly as the anode role is taken over by the peroxide film. After two to three months metallic corrosion ceases almost completely and Figure 1 shows a typical curve at 6 amperes per square foot.

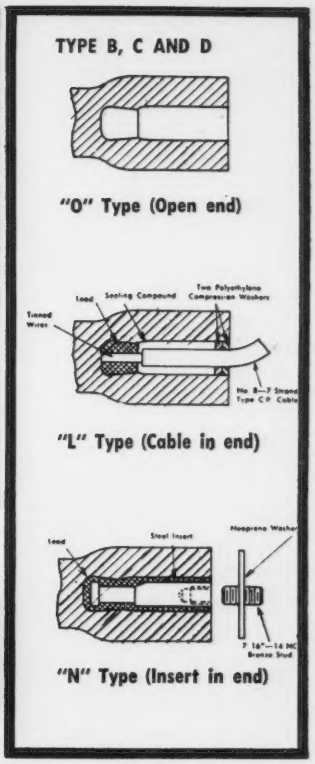
The CX3 alloy can be used successfully at current densities of from 1 to 20 amperes per square foot, this range being the limit of current experimental conditions. Above 30 amperes per square foot blistering of the outer peroxide layer occurs and some shallow pits (20 mils deep after 6 months) develop below these blisters. There is some evidence that lowest consumption occurs at about 10 amperes per square foot, though high performance may be expected between 4 and 20 amperes per square foot. The ease of working and the high density of

(Continued on Page 130)

* By J. H. Morgan, Physicist, Cathodic Corrosion Control, Ltd., London, England.







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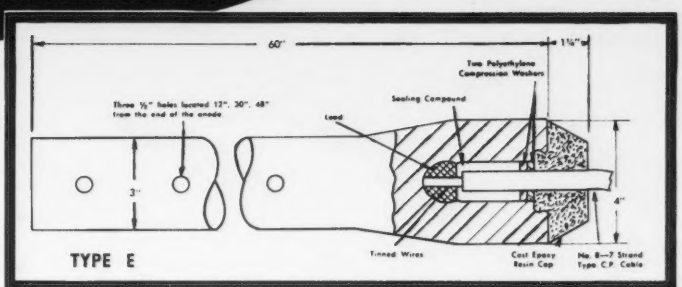
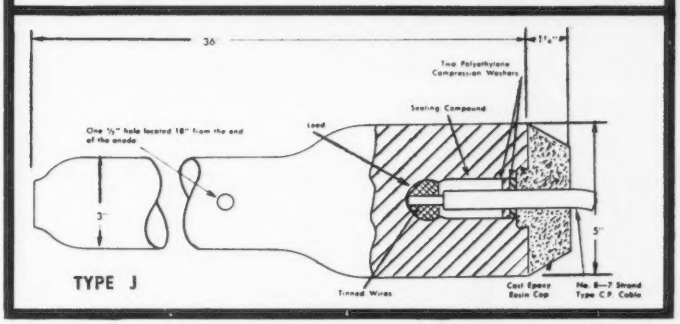
Type C and D — Are used primarily for ground beds. Type C is 1½″ in diameter, 60″ long and Type D is 2″ in diameter, 60″ long.

Type E and J — For salt or brackish water use. Generally used on dock structures, "H" pilings, traveling screens and offshore drilling rigs. Type E is 3″ in diameter, 60″ in length; Type J is 3″ in diameter, 36″ in length.

Type F and FM — Recommended uses include underground ducts, fresh water tanks, and other fresh water type applications where length and flexibility are important.





Type F is 1½″ in diameter and 12″ long; Type FM has 1¼″ diameter and is 9″ long.

Type G — A fresh water anode used primarily for underground ducts, fresh water tanks and other fresh water service where length and flexibility are needed. It is 2″ in diameter and 9″ long.

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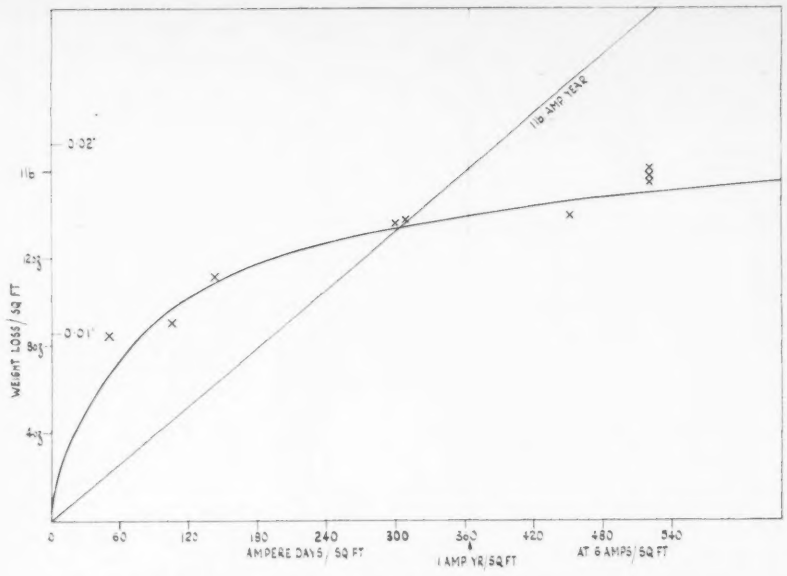

Figure 1—Curve showing consumption of metal from CX3 alloy anodes in sea water.

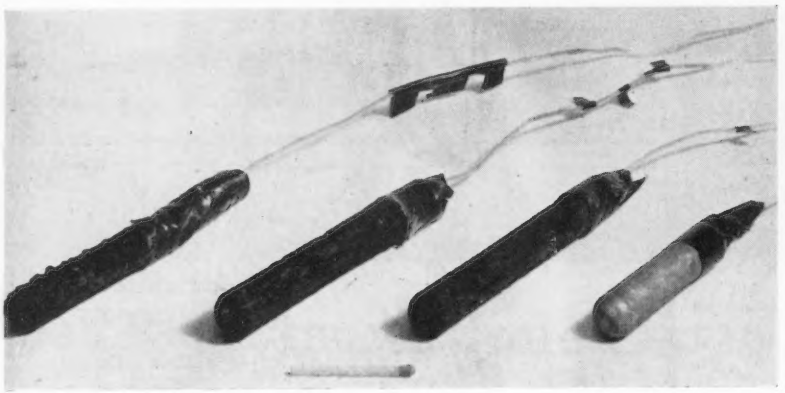

Figure 2—Lead alloy anodes subject to current densities (left to right): 100 amperes sq. ft.; 30 amperes sq. ft. and 6 amperes sq. ft. Anode on right, as cast.

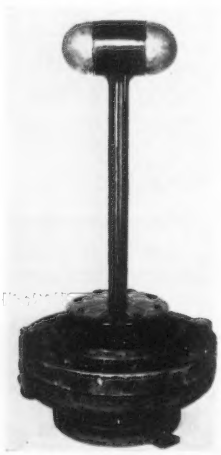

Figure 3—Twin CX3 anodes mounted on insulated support for internal protection of steel pipes carrying sea water. Top flange is mounted on pipe T-junction.

Lead Alloy Anodes–

(Continued From Page 128)

the alloy mean that these current densities are easily realized at low voltages.

Saline Mud and Sands. Anode performance under conditions is almost identical with that obtained in the saturating electrolyte. In both the above environments, in fact in any water whose saline content brings the resistivity below 1000 ohm.cm., the peroxide layer is hard, tough and adheres firmly to the base alloy. Hammering followed by wire brushing is required to remove this film in laboratory determinations.

Fresh Water. Only a limited number of tests have been made because lead seemed unsuitable for potable water. Where fresh water is not used for drinking the driving voltage would be high. Initial tests, however, indicate a performance almost parallel to those of the sea water tests may be expected.

Practical Anodes. Castings and extrusions have been made in this alloy with 1¼-inch diameter extrusions over a tinned copper 37/.083 core.

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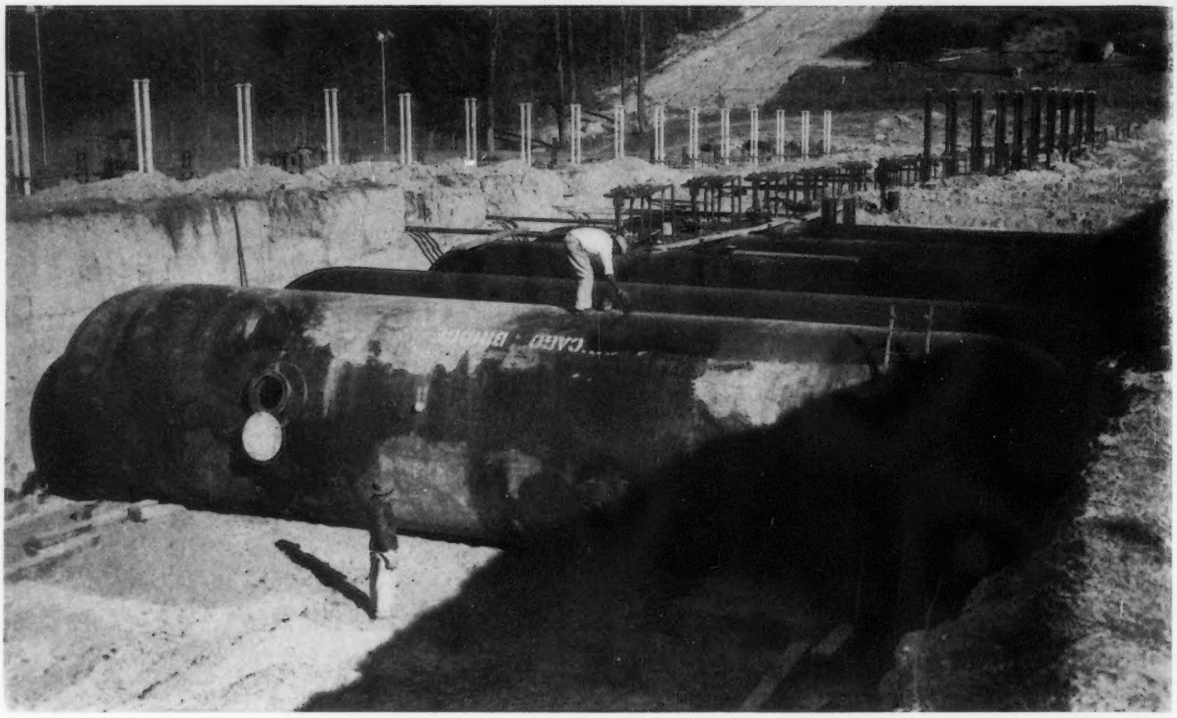

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CORROSION ABSTRACTS

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CORROSION ABSTRACTS

Vol. 13 July, 1957 No. 7

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1. GENERAL

1.2 Importance

1.2.2, 8.9.3, 5.4.5, 5.2.1

Economic Survey of Underground Corrosion Preventive Measures. D. C. GLASS. *Oil in Canada*, **7**, No. 52, 14-17, 27-28 (1955) October 24.

Results of 15 years' experience with underground anticorrosion measures on a trunk pipe-line indicate that cost of cathodic protection for bare 10-inch pipe is less than the cost of reconditioning, coating and supplementing the coat with cathodic protection. Basic economic problems are discussed with particular reference to rectifier installations in the Texas coastal area. Results of a survey of 28 leading pipe-line companies' work 1930-1955 are considered and data for coating practice and cost are tabulated.
11921

2. TESTING

2.3 Laboratory Methods and Tests

2.3.2, 6.2.5, 3.5.8

Stress-Corrosion Cracking of Type 304 Austenitic Stainless Steel. H. L. LOGAN AND R. L. SHERMAN, JR. Paper before Am. Welding Soc., Nat'l Spr. Mtg., Buffalo, May 7-11, 1956. *Welding J.*, **35**, No. 8, 389s-395s (1956) August.

In study of mechanism of stress-corrosion cracking, sponsored by Pressure Vessel Research Council, approximately 40 solutions were tested as possible corrodents to be used in stress-corrosion tests of Types 304 and 304L. Boiling 3½ sodium chloride-1 commercial ammonium nitrate solution, pH 7.0, produced stress corrosion cracking in Type 304 stressed to 80% or more and in the 304L stressed to 100% or more of their room temperature yield strengths. Cracking was produced in 0.2-inch gage lengths strained 0.5% or more. No correlation was found between macroscopic strain and tendency to develop stress-corrosion cracks. Pitting was generally produced by solutions with pH less than 4. Stress-corrosion cracks can develop with little or no previous pitting. Photographs show gas (hydrogen) escaping from cracks. Corrosion products adjacent to cracks contain metal ions in same proportion as parent metal. Test solutions were various concentrations of magnesium chloride, sodium chloride, calcium chloride, ammonium chloride, potassium chloride, ammonium nitrate, zinc chloride, ferric chloride, phosphoric acid and sodium fluoride and sodium chloride in combination with sodium nitrate, potassium chromate, sodium hydroxide, potassium iodide, sodium sulfate and ammonium nitrate. Tables, photomicrographs, 19 references.—INCO.
12899

2.3.4, 2.3.9

Radiochemical Analyses for Iron, Chromium, Nickel and Cobalt Corrosion Products. R. R. SMITH, T. O. PASSELL AND S. D. REEDER. Phillips Petroleum Co. U. S. Atomic Energy Comm. Pubn., AECD-3889, Sept. 9, 1955 (Declassified with deletions January 5, 1956), 14 pp. Available from: Office of Technical Services, Washington, D. C.

Radiochemical and carrier techniques

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BL—Current Technical Literature, Bell Telephone Laboratories, Inc., Murray Hill, N. J.

BTR—Battelle Technical Review, Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio.

CBEC—Centre Belge d'Etude de la Corrosion (CEBELCOR), 17 re des Drapiers, Brussels, Belgium.

CE—Chemical Engineering, McGraw Hill Publishing Co. 330 W. 42nd St., New York 18, N. Y.

EL—Electroplating. 83/85 Udney Park Road, Teddington, Middlesex, England.

EW—Electrical World, McGraw-Hill Publishing Co. 330 W. 42nd St., New York 18, N. Y.

GPC—General Petroleum Corp. of California. 2525 East 37th St., Los Angeles 11, Calif.

IIM—Transactions, The Indian Institute of Metals, 23-B, Notaji Subhas Road, P. O. Box 737, Calcutta, India.

INCO—The International Nickel Co., Inc. 67 Wall Street, New York 5, New York.

IP—Institute of Petroleum. 26 Portland Place, London W#1, England.

JSPS—Japan Society for the Promotion of Science, Address: Mr. Hayata Shigeno, Secretary, Committee of Preventing Corrosion, c/o Government Chemical Industrial Research Institute, 1-Chrome Nakameguro, Meguroku, Tokyo, Japan.

MA—Metallurgical Abstracts, Institute of Metals, London, England. 4 Grosvenor Gardens, London SW 1, England.

MI—Metallurgia Italiana, Associazone Italiana di Metallurgia. Via S. Paola, 10, Milano, Italia.

MR—Metals Review, American Society of Metals. 7301 Euclid Ave., Cleveland 3, Ohio.

NALCO—National Aluminate Corp. 6216 West 66th Place, Chicago 38, Illinois.

NBS—National Bureau of Standards. Supt. of Documents, U. S. Gov't Printing Office, Washington 25, D. C.

NSA—Nuclear Science Abstracts. United States Atomic Energy Commission, Technical Information Division, Oak Ridge, Tenn.

PDA—Prevention Deterioration Abstracts. National Research Council, 2101 Constitution Ave., Washington 25, D. C.

RAD—Reynolds Aluminum Digest, 2500 South Third Street, Louisville, Kentucky

RM—Revue de Metallurgie, Paris, France. 5 Cite Pigalle, Paris (9e), France.

RPI—Review of Current Literature Relating to the Paint, Colour, Varnish and Allied Industries, Research Association of British Paint, Colour & Varnish Manufacturers, London. Waldegrave Rd., Teddington, Middlesex.

SE—Stahl Und Eisen, Verlag Stahleisen, M. B. H., Dusseldorf, August-Thysen Str. 1. Postscheck Koln 4110, (22a) Dusseldorf, Germany.

TIME—Transactions of Institute of Marine Engineers. 85 The Minories, London EC 3, England.

UOP—Universal Oil Products. 310 South Michigan Ave., Chicago, Illinois.

ZDA—Zinc Development Association. 34 Berkeley Square, London W.1.

applied to the determination of minute amounts of corrosion products of Inconel gave results as follows: Iron, 0.061%; chromium, 0.15%; nickel, 0.037% and cobalt, 0.0005%. Step-by-step procedures are given.—NSA. 12976

2.3.4, 4.3.7, 5.4.2, 5.9.2

Detergent Corrosion Test for Vitreous Enamel Surfaces. J. C. HARRIS, M. G. KRAMER AND M. V. TREXLER. *ASTM Bull.*, No. 216, 61-64 (1956) Sept.

Presence of considerable amounts of effective sequestering agents and alkaline builders in synthetic detergent compositions can result in vitreous enamel failure. Description of test for determining corrosive effects of water-soluble cleaners on various vitreous enamel surfaces is given. Stainless steel rack was used for supporting enamel test specimens. Tables give vitreous enamel corrosion test data and visual observation of action of detergents on enamels. Method shows real difference between inhibited and uninhibited compositions. —INCO. 12786

2.3.5, 2.3.7, 5.4.5

Electrochemical Studies of Paint Films on Metals. D. M. BRASHER. *Electroplating & Metal Finishing*, **9**, No. 9, 280-284 (1956) Sept.

Describes in detail three methods developed for the study of paint coatings. Electrode potential measurement is used to investigate the influence of paint thickness, type of finish and pretreatment on life of paint coating. Measurement of electrical resistance and capacitance is used to determine coating life in relation to conditions of application and service, particularly in immersed conditions. Use of capacitance measurement after water takeup determines factors in paint performance and relationship between water uptake and paint life. Use of these methods in evaluating performance of metallic benzoates as inhibitive pigments is described. Graphs.—INCO. 13023

2.3.6

On a New Method of Micrographic Examination of Uranium by the Formation of Epitaxial Layers of Oxide. ANDRE ROBILLARD, JANINE DURAND AND PAUL LACOMBE. *Compt. rend.*, **242**, No. 4, 508-510 (1956).

The examination of α-uranium by polarized light has often been recommended for revealing its structure without attack. The great affinity of uranium for oxygen very considerably limits the efficacy of this method. This reactivity has, however, been put to use to form epitaxial oxide films by a new method of electrolytic etching. After electropolishing the specimen in a bath of chromium trioxide, $C_2H_4O_2$ and water, the potential is lowered rapidly from 30 to 2 or 3 V. without taking the metal out of the electrolyte. As a result an etched structure appears without the use of polarized light by reason of the preferential attack on the grain boundaries, twins, etc. Further, if the metal is kept out of contact of air, it is covered progressively with a film of epitaxial oxide of a thickness detectable by the formation of interference colors. This method enables other observations of the structure of uranium to be made, among the most important of which is the migration of grain boundaries and the detection of certain separate phases.—MA. 12921

2.3.6, 3.5.8, 6.3.6

Interior Deformation Markings in Copper Fatigue Specimens. D. S. KEMSLEY. *Nature*, **178**, 653-654 (1956) Sept. 22.

Microstructures revealed by a technique in which fractured low-stress rotating-cantilever specimens of low-oxygen copper were copper-plated, sectioned diametrically by a slitting wheel flooded with coolant, polished mechanically then electrolytically in phosphoric acid and finally etched in 10 etchants in turn: the most effective etchant was acid ferric chloride. Striations were revealed in the grains.—BNF. 12832

2.3.7

Plating Adhesion: Which Test Do You Buy? P. M. UNTERWEISER. *Iron Age*, **178**, No. 16, 123-125 (1956) Oct. 18.

Discussion of methods of testing plating adhesion. Need for additional research in field of adhesion testing is stressed; most desperately required are tests that lend themselves to mass production techniques while still retaining a high degree of accuracy.—INCO. 1309

2.3.7

A Rapid Method of Estimating the Fatigue Limit or Endurance Limit of Metals in Reverse Bending. J. MCKEOWN. *Metallurgia*, **54**, No. 323, 151-156, 15 (1956) Sept.

Method uses a progressive loading technique. It appears to hold considerable promise, except possibly in the case of materials which are unstable in the conventional fatigue test, e.g., some aluminum alloys. It may have an important application in determining the effect of changes in design in improving or detracting from fatigue performance. (The work described in the paper is based on report R.R.A. 1082 issued to Brit. Non-Ferrous Metals Res. Assoc. members in May, 1955; reprints will be available as R.R.A. 1082P.)—BNF. 12847

2.3.7, 3.2.2

Constant Strain Rate Bend Tests on Hydrogen-Embrittled High Strength Steels. W. BECK, E. P. KLIER AND G. SACHS. Paper before Am. Inst. Mining Met. Engrs., Cleveland, October, 1956. *J. Metals* (Trans. AIME), **8**, No. 10, 1263-1268 (1956) Oct.

Simple bend test was devised in which thin strip is bent as a column. This permits accurate measurement of fracture strains of a steel over wide range of embrittlement conditions. Tensile and bend test results obtained in study of hydrogen embrittlement induced by cathodic treatment in solution of sodium hydroxide are presented. Principal variables which were examined include: current density and time of charging, strain rate and recovery temperature and time. Materials tested were SAE 1070 and SAE 8740. Graphs.—INCO. 13038

2.3.7, 6.3.15

Mechanical Testing of Titanium. Titanium Metals Corp. of America, Titanium Engineering Bull. No. 4, 1956, 16 pp. Also obtainable from British Metal Corp. Ltd., 17 Summer Row, Birmingham, 3.

The methods of test used by TMCA are described in detail: Tensile, hardness, bend and cup, tear, creep, elevated temperature stress stability, notch tensile, room temperature notch stress rupture, impact and fatigue tests; for defects, eddy current and ultrasonic. Actual test results for specific materials are not given, except incidentally for tear load for Ti-75A and stress stability data for Ti-6A1-4V.—BNF. 13096

2.3.9

A New Method for Determination of the Chemical Composition in Alloys. (In Russian.) I. B. BOROVSKII AND N. P. ILIN. Baikov Inst. of Metallurgy. *Doklady Akad. Nauk SSSR.*, **106**, 655-657 (1956) Feb. 1.

A new method and special apparatus were developed for determination of the chemical composition in the micro-volume (~10 μ^3) of alloys using the characteristic radiation excited by focused electron beams. The method enables one to determine various elements in a given point of the microsection and the distribution of certain elements in various areas of it, with the sensitivity up to 0.1%. Experiments made with a series of complex metal alloys, cermets, welded seams and diffusion layers to determine the distribution of iron, nickel, copper, zinc, niobium, molybdenum, tungsten and rhenium elements in microvolumes

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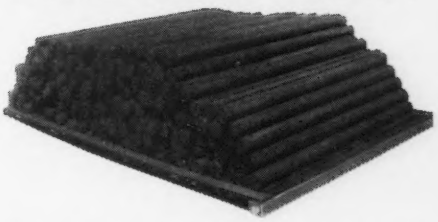

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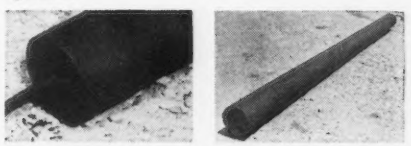

The photographs above illustrate the lead end of one of the older anodes and the bottom half of one of the newer anodes. Original extrusion markings and the rough extrusion end cut are visible.

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133a

and in the given directions, established the practicability of the offered method. —NSA. 13033

2.3.9, 3.8.4, 6.3.6

A New Method of Determining the Thickness of Thin Films Formed on the Surface of Copper by Sulphuration. JACQUES BENARD AND JEAN FRANCOIS LAURENT. *Compt. rend.*, **242,** No. 3, 378-380 (1956).

The estimation of the thickness of thin films of oxides or sulfides formed by reaction at the surface of metals is usually effected by optical, electrometric, or gravimetric methods. In order to obtain very exact and localized measurements, a particularly sensitive method, based on the use of radioactive sulfur, was used. Electron diffraction showed that at the temperature used (350 C), the only compound formed is cuprous sulfide and thus the thickness of the film can be calculated. The results obtained agree very closely with those obtained electrometrically by Dyess and Miley [*Trans. Am. Inst. Mining Met. Engrs.*, **133,** 239 (1939)]. The great interest of this radioactive sulfur method lies in the possibility of effecting localized measurements and in following the growth of a film on crystals of different orientation in the same specimen. The results obtained on crystals in two different specimens are tabulated. It is proposed to study the kinetics of anisotropic growth of thin sulfide films on various metals by this method.—MA. 13029

3. CHARACTERISTIC CORROSION PHENOMENA

3.1 General

3.1, 3.5.8, 1.6

Chemical Engineering Practice. Vol. II. Solid State. H. W. CREMER AND T. DAVIES, Editors. Book, 1956, 661 pp. Butterworths Scientific Publications, 88 Kingsway London, W.C.2.

Contains the following articles of metallurgical interest: Fundamental Concepts of Matter in the Solid State, M. J. P. Musgrave (pp. 1-67); Significance of Mechanical Properties and Their Measurement, P. G. Forrest and M. J. P. Musgrave (pp. 68-112); Alloy Equilibrium Diagrams, R. N. Parkins (pp. 113-143; not a collection of diagrams but indicates principles involved); The Range of Steels, J. D. Gilchrist (pp. 144-175); Fatigue in Metals (pp. 176-208) and Creep in Metals (pp. 209-241), P. G. Forrest; Corrosion of Metals, F. Wormwell and E. Ll. Evans (pp. 255-341); Porous Masses, P. Eisenklam (pp. 342-463; sintered metals, ceramics, etc., their permeability and other characteristics); Porous Powder Metallurgical Products for Filtration and Related Uses, E. Bishop and G. M. Collins (pp. 464-482); Powder Metallurgy, I. Jenkins (pp. 483-532).

Other subjects covered include properties of plastics and glasses and the use of porous solids in water treatment, gas purification, etc. This is one of a series of twelve volumes to be published at intervals.—BNF. 13077

3.2 Forms

3.2.2

Notes on the Brittle Fracture of Steels. W. FELIX AND T. GEIGER. *Sulzer Tech. Rev.*, **38,** No. 1, 14-27 (1956).

Investigation into the mechanism of fracture of steels in the high and low ranges of notch impact values, followed by investigation of an I-girder destroyed by brittle fracture. A 0.05% carbon mild steel and low-alloy steel (0.22 carbon-0.2 silicon-0.99 manganese-0.38 chromium-0.44 copper) were used to study abrupt transition in notch impact value. X-ray diffraction photographs were used to study deformation of crystals in fracture surface of Schnadt's sharp notch specimens; these were compared with photographs of specimens subjected to more or less upsetting. Genesis and development of fracture at notch were studied. A 50-cm I-girder was destroyed by brittle fracture when, after 30 years service, it was dismantled, cut with flame cutter and subjected to 10 C temperature. Fracture was due to pronounced brittleness, combined with structural changes and stresses from flame cutting and with complicated internal stresses in rolled section. Steel composition was 0.09 carbon-0.01 silicon-0.52 manganese-0.058 phosphorus-0.093 sulfur-0.016 nitrogen. Numerous graphs, photomicrographs and X-ray diffraction photographs.—INCO. 12813

3.2.2

Gases in Metals. MANLEY W. MALLETT. *Steel*, **139,** 94-98 (1956) Oct. 8.

Metals were analyzed for oxygen, hydrogen and nitrogen by the vacuum-fusion technique. Embrittlement of various metals is caused by gases which are soluble in these metals in minute amounts. Mechanical, magnetic and ultrasonic methods of vibrating molten metal show promise for degassing.—MR. 12880

3.2.2

Work of the Task Group on Brittle Failure of Steel Forgings. A. O. SCHAEFER. Paper before Am. Soc. Mech. Engrs., Ann. Mtg., Chicago, November 13-18, 1955. *Trans. ASME*, **78,** No. 7, 1623-1626 (1956) Oct.

Covers activities of ASTM Committee A-1, Sub-Committee VI. After brief review of work on importance of hydrogen in steel forgings, reports are presented for three main fields of activity: Testing to appraise tendency of forging to fail in brittle manner; attempt to make turbine or generator rotors less susceptible to brittle failure by heat treatment, by chemistry and by vacuum-pouring technique; and development of methods of inspection and interpretation of inspection to avoid brittle failures. Most common alloy steel used in U. S. is 2.75 nickel-0.50 molybdenum-0.05-0.10 vanadium with less than 0.20 chromium; European grades have lower nickel and higher chromium contents as this typical steel with 2 nickel-1.25 chromium-0.40 molybdenum with or without vanadium. Graph shows temperature transition curve of nickel-molybdenum-vanadium rotor steel based on V-notch Charpy test.—INCO. 12867

3.2.2, 3.5.8

Pitting and Cracking. U. R. EVANS. Paper before Soc. Chem. Ind., Corrosion Group, April 25, 1956. *Chemistry & Industry*, No. 44, 1291-1297 (1956) Nov. 10.

Discussion of causes, spreading and patterns of pitting and cracking corrosion of zinc, iron, aluminum and tin. Pitting and dislocations, intergranular corrosion and weakening, stress corrosion cracking and corrosion fatigue are discussed. Prevention of cracking by protective coatings, heat treatment and peening and nitriding treatments is discussed. 34 references.—INCO. 13021

3.2.3, 6.2.4

The Effect of Nickel Content and Furnace Atmosphere on the Scale Formed on Nickel Steels. K. SACHS. *Metallurgia*, **54,** No. 323, 109-114 (1956) Sept.

Study of the innermost scale layer on nickel steels with results showing the influence of variation of nickel content, the atmosphere in which scale is formed and scale thickness. Steels with nickel contents of the order prevailing in commercial compositions (1½, 3 nickel) were compared with mild steel (no nickel) and a nickel-iron alloy (Nilo 43). Tests were made in controlled and industrial atmospheres. Inner layer of scale contains nickel-rich particles entangled with wustite. Amount of metal in scale diminishes with increasing partial pressure of oxygen in the oxide and with falling nickel content. Furnace atmosphere and scale thickness determine partial oxygen pressure at various levels in the scale and thus the nickel content of the metal phase. In tests with a 3 nickel-0.09 antimony steel, it was found that antimony accumulated in those fractions of scale which had been enriched in nickel in same proportions as the nickel. Numerous photomicrographs, graphs.—INCO. 12923

3.3 Biological Effects

3.3.2, 5.4.8

Anti-Fouling Paints Progress. Part II. J. C. KINGCOME. *Paint Manuf.*, **26,** 206-210 (1956) June.

Some theories on the mechanism of action of anti-fouling paints. Future developments in the field. Photographs. 16 references.—BTR. 12874

3.3.3, 6.3.8

Cause of Acid Leakage in Lead Chambers. (In French.) A. VALLAND AND P. SALMON. *Corrosion et Anticorrosion*, **4,** No. 4, 135-138 (1956) April.

Small holes formed in lead vessels designed to hold sulfuric acid or other acids were found to be due to an insect *(Sirex)* boring from outside. Methods of protection, by insecticides or heating to 100 C, are suggested.—BNF. 13118

3.3.3, 7.7, 6.3.8

Marine Borer Attack on Lead Cable Sheath. L. R. SNOKE AND A. P. RICHARDS. *Science*, **124,** No. 3219, 443 (1956) Sept. 7.

Documents recent case in which a mollusk penetrated a lead-sheathed submarine telephone cable. Cable lies in Ortega River, Jacksonville, Florida. 4-mm thick lead sheath was covered with two layers of asphalt-impregnated jute which serve as bedding for single layer of galvanized steel armour wire. Attack occurred 350 feet from shore 3.5 feet deep in mud. Dimensions of holes suggest they were caused by member of *Pholadidae* family. Holes in sheath are described and illustrated.—INCO. 13092

3.5 Physical and Mechanical Effects

3.5.8

Variation in Fatigue Properties Over Individual Casts of Steel. Pt. I. Investigation of a Cast of Steel to B.S. 970 Specification En. 100. E. INESON, J. CLAYTON-CAVE AND R. J. TAYLOR. Brit. Iron and Steel Res. Assoc. Paper MG/M/74/56

of Fatigue and Inclusions Committee. *J. Iron Steel Inst.*, **184,** Pt. 2, 178-185 (1956) Oct.

Investigation to determine whether or not fatigue properties vary within individual ingot of acid O.H. steel made to S.S. 970 En. 100. 500 test pieces, hardened and tempered to U.T.S. of 60-65 tons/in², were tested in fatigue according to a statistically designed experiment. Composition variations, distribution of non-metallic inclusions, hardenability and tensile properties are considered. Longitudinal fatigue properties are relatively uniform throughout. Only noticeable variation occurred at top of ingot which exhibited slightly better properties. Dispersion of non-metallic inclusions was uniform throughout and fracture surfaces showed no evidence of inclusions being responsible for initiation of fatigue failures. Tables, graphs.—INCO. 12781

3.5.8

The Effect of Stress on Microhardness. F. H. VITOVEC. University of Minnesota. U. S. Wright Air Development Center Pubn., August, 1956, 28 pp. Available from: Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 121534).

An investigation of stress effects on the microhardness of mild steel, brass and copper showed that for initially stress free specimens, tensile stress decreased microhardness over a certain range of test load and compressive stress caused a slight increase. As a function of test load for mild steel, Armco iron, copper, brass, zinc and cadmium, microhardness revealed a discontinuity of slope in the hardness-load curve at light loads. Residual stresses introduced by the polishing method can significantly affect the trend of the hardness-load relationship, results of the research indicate. 13095

3.5.8

Surface Film Formation and Metallic Wear. W. HIRST AND J. K. LANCASTER. *J. Applied Physics,* **27,** No. 9, 1057-1065 (1956) Sept.

Experimental study of way in which wear is affected by factors known to influence friction. When a relatively soft metal slides on a harder one, relationship between wear and sliding distance may be 1 of 3 general types, each of which is associated with the formation of a surface film during sliding. Extent to which these films prevent intermetallic contact influences relationship between wear rate and applied load. With several metals there is a discontinuity in the wear rate-load relationship and two distinct regimes of wear are obtained. Transition between these two regimes is associated with breakdown of a protecting surface film. Graph showing wear rate with load for various metals rubbing on Stellite and lubricated by cetane includes steel, stainless steel, aluminum and iron. Illustrations.—INCO. 13061

3.5.8, 3.7.4

Structure Dependent Chemical Activity of Polycrystalline Cu_3Au: Experiments Relating to the Mechanism of Stress-Corrosion Cracking of Homogeneous Solid Solutions. R. BAKISH AND W. D. ROBERTSON. Paper before Am. Inst. Mining Met. Engrs., Cleveland, October, 1956. *J. Metals* (Trans. AIME), **8,** No. 10, 1277-1282 (1956) Oct.

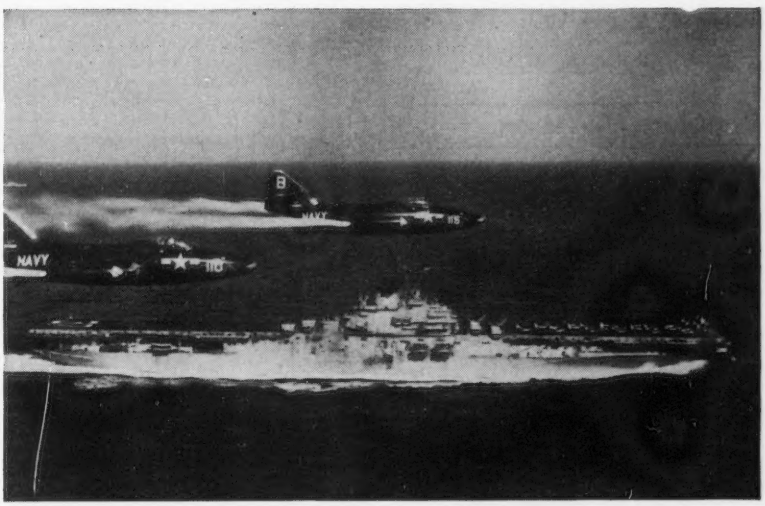
To provide information necessary for more general explanation of stress corrosion cracking in homogeneous solid solutions, following structural factors were investigated in a 48.92 copper-51.01 gold alloy (Cu_3Au); effect of long and short order range, structural sites from which copper is removed from annealed and cold worked polycrystalline alloy and the effect of annealing time and temperature on structural reactions in cold worked alloy. Corrosive medium was ferric chloride solution. Copper is selectively removed from alloy at grain boundaries, incoherent twin boundaries and deformation bands, irrespective of the state of order. Galvanic potential developed between alloy and remaining gold supplies driving force for continued penetration in direction defined by structural discontinuities. Graphs, photomicrographs.—INCO. 13055

3.6 Electrochemical Effects

3.6.8, 6.4.2, 3.8.2

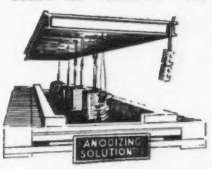
Hydrogen Overpotential on Aluminum. D. J. HANSEN AND F. E. W. WETMORE. *Can. J. Chem.,* **34,** No. 5, 659-664 (1956) May.

The hydrogen overpotential of 1) rolled sheet 99.995% pure, 2) cast polycrystalline, 99.995% pure, 3) single crystal, 99.995% pure, 4) "commercially pure" ingot, 99.5% pure aluminum in 0.1 normal sulfuric acid at 25 C was studied. The aluminum cathode was electropolished in a perchloric acid bath, then mounted in a Teflon box which exposed only one flat face. The aluminum cathode and platinum anode were in separate compartments of the cell. Rates of



CHROMIUM CHEMICALS IN ACTION

The useful life of modern jet aircraft has been appreciably prolonged through the application of effective corrosion inhibitors. Chromium Chemicals in various forms are used at many stages of jet plane production.



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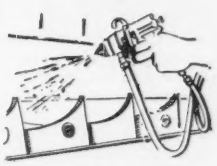

These illustrations are examples of the ability of Chromium Chemicals to inhibit and prevent corrosion. Besides supplying many industries with superior quality chromium chemicals, Mutual offers information on the numerous other ways that these chemicals help to protect valuable materials and equipment. So, if you are in doubt about the best way to solve your corrosion problem, contact Mutual.

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corrosion were determined by measurement of the weight loss of the sample. Curves of the overpotential plotted against the logarithm of the applied current density show a distinct deviation from the linearity frequently found for unreactive metals, expressed by Tafel's relation: $\eta = b \ln (i/i_o)$. Even when there is no applied current there is a cathodic current due to the auto-corrosion of the sample. With the exception of sample 4), the cathode showed a decrease in the slope of the "Tafel curve" at the higher current densities. This may be attributed to an alteration in the nature of the active cathodes. It is assumed that both the aluminum and the traces of impurities present can act as cathodes. The effect of the autocorrosion is interpreted and the role of impurities in determining the overpotential and the rest potential is shown. In addition build-up of decay of the overpotential was studied. Its behavior is more complicated than that of noble metals.—ALL. 11989

3.7 Metallurgical Effects

3.7.3

The Effect of Aluminum Additions to Mild-Steel Weld Metal. C. R. SIBLEY. Paper before Am. Welding Soc., National Fall Mtg., Philadelphia, October 17-21, 1955. *Welding J.*, **35,** No. 8, 361s-368s (1956) August.

When mild and low-alloy steel electrodes, used in gas-shielded arc welding, are coated with aluminum or aluminum-silicon alloy, advantages achieved are improved corrosion resistance of electrode during storage and elimination of weld-metal porosity because large amount of aluminum is available for deoxidation. Samples of aluminum-coated steel wire welding electrodes were prepared for evaluation and comparison with bare and copper-flashed electrodes. Effect of coatings on welding characteristics and mechanical properties of weld metal are presented. Impact test results are plotted down to —80F. Tables, graphs, photomicrographs.—INCO. 12996

3.7.3

Wettability of Steel with Pure Silver and Various Silver-Brazing Alloys. N. BREDZS. Paper before Am. Welding Soc., Nat'l Fall Mtg., Philadelphia, October 17-21, 1955. *Welding J.*, **35,** No. 10, 508s-520s (1956) Oct.

Experimental results obtained on the investigation of wettability of low-carbon steel with pure silver and various silver base alloys proved that incomplete protection against exposure to air might considerably impair wettability. Times as short as ½ second are sufficient to cause dewetting by molten filler metals. Study was made of influence of small addition of various metals on diffusion of oxygen through layer of molten silver. Nickel and tin may completely protect molten silver from oxygen of the air. Metals with highest affinity for oxygen (lithium, aluminum, magnesium, barium, etc.) are not able to prevent or slow down oxygen diffusion. Small additions of copper accelerate the diffusion. Additions of lithium and barium improve wettability of liquid silver on steel by reducing iron oxides at practical brazing temperatures. Nickel or tin additions can be used for self-fluxing silver-brazing alloys to protect against oxygen diffusion. Photomicrographs.—INCO. 13021

3.7.3

Methods of Joining Non Ferrous Metals. (In German.) ALEXANDER MATTING. *Z. Metallkunde,* **47,** No. 7, 445-447 (1956) July.

Physical and chemical problems connected with the buildup of a sprayed metallic layer are analyzed and some practical applications of this process, such as the metallizing of bearing surfaces, are described. A sketchy treatment of the corrosion behavior of light metal weld beads, subjected to both chemical and electrolytic attack, is presented and some aspects of the adhesive bonding techniques applied in combination with conventional joining methods in particular are briefly dealt with.—ALL. 12843

3.7.3

The Welding and Soldering of Non-Ferrous Metals. (In German.) *Z. Metallkunde,* **47,** No. 7, 445-516 (1956) July.

The major part of issue devoted to welding or soldering of non-ferrous metals: Jointing Methods for Non-Ferrous Metals, A. Mattig (pp. 445-447. Welding of non-ferrous metals; flame spraying of e.g., bearings; corrosion behavior of light metal welds; adhesive bonding); Common Arc-Welding Processes, O. Becken (pp. 448-452. Customary processes and their field of applications); Spot Welding of Aluminum Alloys, L. Hutten and E. A. Otto (pp. 453-458. A survey (20 references) of defects arising during spot welding aluminum and suitable welding conditions); Inert-Gas Welding of Copper and Its Alloys, L. Wolff and W. Mantel (pp. 459-465. Description of equipment and technique for argon arc and Sigma welding of electrolytic, tough pitch deoxidized and OFHC copper, brass, aluminum brass and bronze, bronze, tin bronze, silicon bronze, lead bronze, copper-beryllium, copper-chromium and copper-cadmium); (Present) Status of Magnesium Welding, E. Rubo (pp. 466-473. Survey of available methods and their fields of application. 10 refs.); Testing and Evaluation of Gas Welded Joints in Zinc and Zinc Alloys, N. Ludwig (pp. 473-477. Development and application of testing methods with some reference to the German literature. Examples are gas welded sheets of pure zinc (99.99), commercial zinc (98.5), zinc-lithium, zinc-copper (1.2 and 4% copper), zinc-aluminum (1 and 10% aluminum) and zinc-10 aluminum-1 copper. 8 references); Progress in Fusion Welding of Non-Ferrous Metals and Alloy Steels, R. Dumpelmann (pp. 478-486. A survey, no references. Effect of composition of filler wire for welding aluminum, copper, nickel and their alloys, cast iron and alloy steels); Behavior of Welded Light Metal of the Type AlZnMgl, A. Muller-Busse (pp. 486-490); The Wetting Power of Solders, A. Keil (pp. 491-493. Wetting power of soft solders and brazing metals; wetting of ceramic materials by molten metals); Electric Resistance Furnace Brazing in a Protective Atmosphere, G. Simon (pp. 494-499. Design of parts for brazing and description of various furnaces and associated equipment); Welding Filler Metal and Brazing Metals of Copper and Copper Alloys, H. Klein (pp. 499-506. A survey of composition, physical and mechanical properties and application of copper-base alloys for filler metals for welding and as brazing alloys); Some Problems in the Production of Structural Metal Joints with Synthetic Resin Adhesive, K. Meyerhans (pp. 506-516. Composition, properties and advantages of some adhesives for the aircraft industry, rockets, car brakes, transformers, etc.)—BNF. 13043

3.7.3, 2.4.2

The Origin and Distribution of Hydrogen in Steel Weld Metal. E. J. MILLETT. Brit. Welding Res. Assoc. Report No. FM 3/99/56. *Brit. Welding J.*, **3,** No. 10, 497-509 (1956) October.

Available data on mechanism of absorption and retention of hydrogen by steel weld-metal are reviewed. It is concluded that there is significant segregation of the gas into diffusible hydrogen which is evolved at room temperature and hydrogen retained in molecular form in voids. Conditions necessary for experimental determination of segregation of hydrogen in weld metal are deduced. Apparatus for determination of diffusible and retained hydrogen contents of small weld metal deposit immediately after welding are described. Intermediate segregation of hydrogen at lattice faults, suggested by some hydrogen embrittlement theories, is not determined separately by extraction. 82 references.—INCO. 12841

3.7.3

Welding and Brazing Refractory Metals: Molybdenum, Niobium, Tantalum, Zirconium. F. G. Cox. *Murex Review,* **1,** No. 16, 429-460 (1956).

A thorough survey. Homogeneous welding; zirconium, tantalum, and niobium (argon arc welding; microstructure; corrosion resistance; tensile properties; application; argon arc spot welding; resistance welding); niobium (argon arc welding, resistance welding; pressure and upset welding). Brazing and soldering; zirconium, tantalum, niobium, molybdenum. 14 references.—BNF. 11960

3.7.3

Effect of Rolled and Annealed Surface Layer on Corrosion of Steel and the Service Life of Protective Coatings. (In German.) FRANZ EISENSTECKEN. *Arch. Eisenhuttenw.*, **27,** No. 3, 179-185 (1956) March.

Role of the above surface layer in aqueous solutions and in steam boilers. Table, graphs, diagrams, photographs. 11 references.—BTR. 12016

3.7.3

Corrosive Fluxes: Their Role in Soldering. R. M. MACINTOSH. Paper before Am. Soc. Testing Materials, 59th Ann. Mtg., Atlantic City, June 17-22, 1956. ASTM Preprint No. 89b, 1956, 4 pp.

Reviews classification of fluxes, gives compositions of corrosive flux types suitable for joining common metals and alloys and discusses application and removal of flux residues.—INCO. 12118

3.7.3, 6.4.2

Fluxless Aluminium Joining Avoids Joint Corrosion. S. FREEDMAN. *Iron Age,* **177,** No. 9, 71-73 (1956) March 1.

Recommends use of a zinc-lead alloy (85-90% zinc, 1.4-7% lead, 0.6-3.0% tin, 0-8% aluminum, 0-5% copper) for joining aluminum castings, sheet or foil: addition of hydrochloric acid via carrier said to promote miscibility of lead in zinc; solder is said to be self-fluxing and presence of aluminum oxide on the surface of aluminum or aluminum alloy does not affect the soldering. These solders are also claimed to be suitable for zinc and alloys, galvanized or aluminized sheet, soldering copper to aluminum, etc.—BNF. 12023

3.7.3

Properties of Arc-Welded Joints Between Aluminum and Stainless Steel. M. A. MILLER AND E. W. MASON. Paper before Am. Welding Soc., Nat'l Spring Mtg., Buffalo, May 7-11, 1956. *Welding J.*, **35**, No. 7, 323s-328s (1956) July.

Describes proper method of joint preparation and design and summarizes available data on performance of metallurgically bonded aluminum-steel joints. Particular attention is directed to inert-gas-shielded tungsten-arc-welded joints between aluminum and aluminum-coated stainless steel tubes. Effect of postheating on shear strength of aluminum-steel joints indicates that welded tube joints may operate safely at temperatures up to 500 F for long periods without loss of shear strength. Thermal shock cycling tests at room temperature—500 F on brazed aluminum Type 347 joints suggest that arc welded joints will withstand this treatment without damaging effect. Results are given of corrosion tests of arc-welded joints. Though exposure to hot tap water resulted in severe pitting and high loss of bursting strength, 6 months' exposure to standard salt spray test was virtually without effect. Tables, graphs, photomicrographs. —INCO. 12537

3.7.3, 7.7, 6.3.6, 6.4.2

Welding Aluminum to Copper Using Inert-Gas Metal-Arc Process. L. A. COOK AND M. F. STAVISH. Paper before Am. Welding Soc., Nat'l. Fall Mtg., Philadelphia, October 17-21, 1955. *Welding J.*, **35**, 348-355 (1956) April.

Increasing use of aluminum busbar at elevated temperatures in combination with copper bus systems increased need for method of arc welding aluminum to copper. Portion of copper to be joined is coated with silver solder, then aluminum and copper are welded using consumable-electrode inert-gas metal-arc method. Mechanical, electrical and metallurgical characteristics of various designs are detailed. In corrosion tests at Daytona Beach, samples were placed on racks so that copper member was on top, permitting drainage of copper corrosion products down over aluminum, the most corrosive condition. After a year, entire sample, including aluminum suffered only superficial corrosion with no measurable change in electrical resistance of joints. Photograph shows test racks.—INCO. 11959

3.7.3, 6.3.20

Embrittlement of Multipass Welds in Zircaloy 2. J. T. NIEMANN AND R. P. SOPHER. *Welding J.*, **35**, No. 1, 27s-31s (1956) Jan.

Bend ductility and tension-impact strength of welds in Zircaloy 2 (zirconium-1.55 tin-0.1 chromium-0.12 iron-0.05 nickel) are lowered by precipitation of intermetallic compounds in the welds but can be restored by suitable post-weld heat-treatment.—BNF. 12102

3.7.3

Effect of Fluxes on Soldered Connections. A. Z. MAMPLE. *Electrical Mfg.*, **57**, No. 2, 124-128 (1956) Feb.

By evaluation of 20 samples the author claims to have established the superiority of water-white rosin flux for electrical and electrolytic wire joints. The chief advantages claimed over activated and proprietary fluxes include a low melting point, possession of a controlled natural acid and a flux residue that is non-conductive, non-corrosive and non-hygroscopic. Includes some brief notes on soldering of a variety of metals with rosin fluxes.—BNF. 12106

3.7.3, 5.3.2

The Welding of Galvanized Steel: A Review of Recent Literature. P. W. JONES. *Welding and Metal Fabrication*, **24**, No. 3, 98-101 (1956) March.

Welding techniques, strength and corrosion resistance of the welds, "regalvanizing" methods (Galv-Weld, Armco Brand E., aluminum paints, zinc-rich paints, flame spraying), fume hazards and ventilation. 48 references.—BNF. 12044

3.8 Miscellaneous Principles

3.8.4

Composition of Photo-Active Particles, Liberated by Metals in Atmospheric Corrosion. (In Russian.) I. L. ROIKH. *Doklady Okad. Nauk SSSR*, **108**, No. 6, 1102-1105 (1956) June.

Reactions of zinc and hydrogen peroxide vapor.—BTR. 12864

3.8.4

Initial Oxidation Rate of Metals and the Logarithmic Equation. HERBERT H. UHLIG. *Acta Metallurgica*, **4**, No. 5, 541-554 (1956) Sept.

The logarithmic oxidation equation $y = k_o \ln (t_r + 1)$ is derived assuming control of the rate by electron flow from metal to oxide. Electron flow during oxidation, in turn, is a function of a changing less-positive or a more-negative space charge in the oxide extending up to several thousand angstrom units from the metal surface, where increasing numbers of trapped electrons at lattice defect sites account for the changing space charge. The space charge is composed of two parts: 1) a uniform-charge density layer next to the metal and 2) a diffuse-charge density layer beyond the uniform layer. Oxidation follows the logarithmic equation during formation of both space-charge layers, but not afterward, with a higher oxidation rate accompanying formation of the diffuse layer. It is shown that under some circumstances the particular distribution of negative charge in the diffuse layer may lead to the cubic oxidation equation. Experimental conditions, especially impurities present in the oxide and its heat treatment, probably determine which electric-charge distribution is favored. For oxidation beyond the space-charge layer, either the linear equation is obeyed, with control of the rate still focused at the metal-oxide interface or, more frequently, the parabolic equation is obeyed with control centered in diffusion and migration processes in the oxide, in accord with Wagner's theory. From oxidation data for copper, the density of trapped electrons in the uniform charge density cuprous oxide layer at 150C is calculated to be 1.3×10^{15}, which decreases to 6×10^{13} at 250C. The number of available sites for trapped electrons in the diffuse layer is calculated correspondingly to be 1.0×10^{14} and 2.6×10^{12} respectively. The thickness of the uniform charge density layer varies from 360 Å at 150° to 1630Å at 250C. The Rideal-Jones empirical relation $\Delta E = \phi - 3.6$, where ΔE is the activation energy for oxidation and ϕ the metal-work function, both in electron volts and which has been shown to hold for carbon, tungsten, and platinum, is shown herewith to apply to nickel, tantalum and titanium and more approximately to copper and iron. This relation is derived

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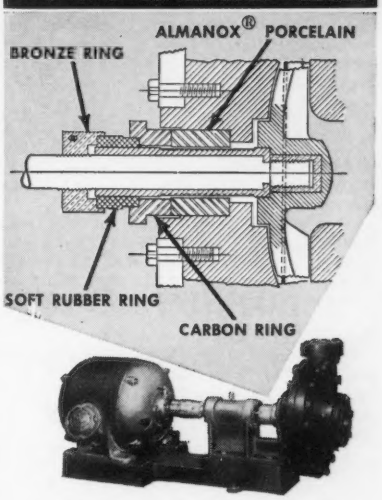

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theoretically from the same fundamental assumptions used in deriving the logarithmic oxidation equation. (auth)—ALL. 13043

3.8.4

Marker Movement in Scale Layers. K. SACHS. *Metallurgia,* **54,** No. 321, 11-17 (1956) July.

Nickel and copper markers in the form of electrodeposits and wires of various thickness were placed on the surface of mild steel specimens which were subsequently oxidized at 900C. It was found that thick wires moved outward in the scale during oxidation and thin markers inward (intermediate sizes occupying intermediate positions). Copper markers moved further inward than nickel for corresponding size. Possible mechanisms are discussed.—BNF. 12956

3.8.4, 6.3.11, 3.2.2

A Study of Internal Oxidation of Alloys of Silver with Cadmium and Copper. (In Russian.) V. V. USOV AND E. M. MURAV'EVA. *Fizika Metallov i Metallovedenie,* **2,** No. 3, 552-561 (1956).

An experimental study of the time and temperature dependence of the process of internal oxidation in alloys annealed in oxygen and the effect of oxygen pressure on that process.—BTR. 13076

3.8.4, 6.3.11, 4.3.5

Continuous Recording Optical Study of the Corrosion of Silver by Iodine Vapour. (In French.) GEORGES COLANGE AND RENE DUBRISAY. *Compt. rend.,* **242,** No. 3, 363-365 (1956) Jan. 19.

Colange and Dubrisay previously studied [*ibid.,* **239,** 1629 (1954)] the surface corrosion of silver by iodine vapor by continuous recording of the interference rings on a specimen pierced by a hole. In order to avoid the disturbances caused by the passing of the vapor over the hole, they sought to set up an ideal case by placing a silver disc in a tightly closed box itself placed in the receptacle containing the iodine, the whole being enclosed in glass. This is held at a const. temp., iodine-saturated atmosphere is admitted and the lid of the box then rapidly lifted. The recording apparatus used was that previously described. The attack was more rapid than in the earlier experiments. Curves of the thickness of the iodide layer against the corresponding time were drawn for different places on the disc, the temperature being 42C. From the second interference ring onwards the curve is linear but the rate of attack at the origin appears to be very high. Experiments at other temperatures between 30 and 70C were made and a curve was drawn of the slope of these curves against the temperature. Up to 44C this curve is linear; above this the variation is also linear but slower.—MA. 13015

4. CORROSIVE ENVIRONMENTS

4.2 Atmospheric

4.2.1

Atmospheric Corrosion of Metals. Part I. B. SANYAL AND G. K. SINGHANIA. *J. Sci. Ind. Research* (India), Sec. B. **15,** 448-455 (1956) August.

A correlation was observed between rates of corrosion and degree of atmospheric pollution and meteorological factors for mild steel and zinc.—BTR. 12920

4.2.3

Flue Gas Solves Lime Waste Treatment Problems. M. D. GIVEN. Eastman Gelatine Corp. *Power Eng.,* **60,** No. 10, 93-98 (1956) Oct.

Description of flue-gas method of precipitating heavy lime wastes and scrubbing flue gas with soda ash to remove both fly ash and corrosive sulfur compounds. Scrubber designed to remove fly ash and corrosives sufficiently to permit flow of gas reduced corrosion of steel pipelines, blowers and other equipment. Photos, diagrams.—INCO. 12794

4.2.3, 4.3.3

The Fouling of Turbine Blades by Fuel Ash. Pt. V. Corrosion Tests with Various Heat Resisting Materials. F. S. E. WHITCHER. Gt. Brit. Ministry of Supply, Nat. Gas Turbine Estab., NGTE Rept. No. 192, June, 1956, 25 pp.

Investigation of resistance to oxidation by fuel ash containing vanadium pentoxide employing series of rig tests which provide continuous deposition of fuel ash. Rig is hydrogen cyclone type combustion chamber fitted with rectangular test duct. Gas temparture is 1150 C, and air cooling injected between stages gives blade temperatures of 800-600 C. Admiralty reference fuel "Mothball" with and without magnesium addition (to form magnesium naphthenate) was used. Materials tested included nickel base alloys Nimonics 75, 80, 80A and 90, and ERA ATG; cobalt-base alloys G.32, Vitallium and modified Vitallium; and iron-base alloys G18B, Rex 337A, HGT3, Pyrista, Immaculate 5, ERA ATV, H40, H46, FCBT, R20, R22, Rex 326F and H.R. Crown Max. Also tested were nickel, sintered titanium carbide, nickel plating and stonecladding. Nimonic 80A blade was used as the control. Nickel- and cobalt-base alloys were more resistant than iron-base alloys. Tables, graphs.—INCO. 13109

4.3 Chemicals, Inorganic

4.3.2

Properties of Fuming Nitric Acid Affecting Its Storage and Use as a Rocket Propellant. D. M. MASON. Paper before Am. Rocket Soc., Ann. Mtg., Chicago, November 14-18, 1955. *Jet Propulsion,* **26,** No. 9, 741-744, 756 (1956) Sept.

Some of the general problems of storing fuming nitric acid (the system nitric acid-nitrogen peroxide-water) in closed metal containers for months at elevated temperatures (130 F) are discussed. Corrosion of aluminum alloys and 18-8 by fuming nitric acid is inhibited in both liquid and gas phase by 0.4-0.6 hydrofluoric acid; passivation mechanism is formation of impervious coating of insoluble metal fluoride. Problem of thermal decomposition and excessive storage pressures is eliminated in acid containing 12-16 nitrogen peroxide and 2-3.5 water. Charts summarize corrosion data for aluminum alloys, stainless steels, 17-7PH, 19-9DL, 19-9 DX, chromium, Ni-A, tantalum, titanium and lead. Graphs, 29 references.—INCO. 12845

4.3.2, 5.8.2, 5.9.4

Linearly Selective Corrosion of Nitrided Steel by Sulfuric Acid Containing Admixtures. (In Russian.) S. A. BALEZIN AND V. B. RATINOV. *Doklady Akad. Nauk SSSR,* **108,** No. 5, 903-904 (1956) June 11.

Distinguishes betwen additives to sulfuric acid which affect primarily the cathodic regeneration of hydrogen ions and those of primarily anodic action and finds that best corrosion inhibiting results are achieved by simultaneous use of additives of both types.—BTR. 13016

4.3.3

Corrosion of Metals in Uranyl Nitrate Solution. N. GOLDOWSKI *and* L. RUCKER. Chicago Univ. U. S. Atomic Energy Comm. Pubn., CT-3152, July 19, 1945 (Declassified Jan. 30, 1956), 9 pp. Available from: Office of Technical Services, Washington, D. C.

Data on the corrosion of aluminum, beryllium and niobium at 90 C by aqueous solutions containing up to 4 gm uranium/liter and up to 1 N nitric acid are presented. Aluminum was seriously attacked, whereas niobium was very resistant during 15 days' exposure, even in the presence of 10^{-3}N hydrogen peroxide. Beryllium was attacked to an important extent (1.5 mg/cm² in 5 days) in 0.1 N nitric acid and more rapidly in higher concentrations of nitric acid. (auth).—NSA. 12808

4.3.3.

Corrosion of Metals in Uranyl Sulfate Solution. N. GOLDOWSKI, W. CARLSON AND L. RUCKER. Chicago Univ. U. S. Atomic Energy Comm. Pubn., CT-3153, July 19, 1945 (Declassified Jan. 30, 1956), 10 pp. Available from: Office of Technical Services, Washington, D. C.

Lead is seriously attacked at 100 C in 0.1 N sulfuric acid containing up to 4g uranium/liter as uranyl sulfate, the attack increasing with the uranium concentration. In the range 0.5 to 1.0 N sulfuric acid, the corrosion product accumulated on the specimen. A protective film appeared to form on lead in 1 N sulfuric acid at 70 C during a 15-day exposure. A somewhat thicker film was formed when the solution also contained 10^{-3}N hydrogen peroxide. Niobium was very resistant to corrosion under the conditions studied. (auth.)—NSA. 12804

4.3.3, 4.2.3

Vanadium Pentoxide Corrosion of Metals at High Temperatures. Part II. Behavior of Some metals (Iron, Cobalt, Nickel, Chromium) and Their Alloys. (In Italian.) A. BURDESE AND C. BRISI. *Metallurgia Italiana,* **48,** 349-357 (1956) August.

On basis of results obtained with systems involved vanadium, oxygen and nickel, cobalt, chromium or iron, research was carried out to identify compounds in corrosion products formed when nickel, cobalt, chromium, iron and their alloys are attacked by vanadium pentoxide.—INCO. 13030

4.3.3, 4.3.4

Corrosion of Aluminum and Beryllium in Ammonium Carbonate-Uranyl Nitrate Solution. N. GOLDOWSKI AND L. BEAMON. Chicago Univ. U. S. Atomic Energy Comm. Pubn., CT-3151, July 19, 1945 (Declassified Jan. 30, 1956), 5 pp. Available from Office of Technical Services, Washington, D. C.

Aluminum and beryllium were separately exposed at 70 C in 0.5 M ammonium carbonate solution containing 4g uranium/liter added as uranyl nitrate. Neither metal was seriously attacked during a 10-day exposure. An eight-hour exposure in a similar solution which contained up to 0.5 N hydrogen peroxide greatly accelerated the rate of corrosion of aluminum but had no effect on beryllium. (auth.)—NSA. 12806

4.3.5

Diaphragm Materials for Electrolytic Fluorine Cells (Problem Report). J. C.

SMITH, E. I. Du Pont de Nemours and Co. U. S. Atomic Energy Comm. Pubn., M-3029, June 28, 1943 (Declassified January 13, 1956), 6 pp. Available from: Office of Technical Services, Washington, D. C.

The corrosion resistance of various materials to fluorine was tested.—NSA. 13122

4.3.5

Plant for Packaging Fifteen Pounds C-216 (Fluorine) per Day by the Liquefaction Method (Problem Report JWD-45). J. F. FRONING AND M. K. RICHARDS. Du Pont de Nemours (E. I.) and Co. U. S. Atomic Energy Comm. Pubn., M-2557, Jan. 20, 1945 (Declassified Jan. 13, 1956), 16 pp. Available from: Office of Technical Services, Washington, D. C.

Design features are described of facilities for packaging approximately 15 lb/day of fluorine. The physical and chemical properties of the gas demand extreme attention to details of construction, operation and maintenance. These details are discussed.—NSA. 12810

4.4 Chemicals, Organic

4.4.1, 4.6.2, 4.3.6, 4.7

Corrosion by Heat Exchange Liquids and Refrigerants. (In French.) M. J. BIGEON. *Corrosion et Anticorrosion*, **4**, No. 4, 139-145 (1956) April.

Applicable temperature ranges and corrosive effects of saturated water vapor, aqueous solutions of salts, superheated steam, chlorinated hydrocarbons, ethylene glycol, mineral oils, organic silicates, aromatic hydrocarbons, molten salts. 13 references.—BNF. 13126

4.4.3, 5.8.2, 4.6.4

Automotive Antifreezes. F. L. HOWARD, B. BROOKS AND R. E. STREETS. U. S. National Bureau of Standards Circular No. 576, July, 1956, 23 pp. Available from: Superintendent of Documents, Washington 25, D. C.

General survey based on 191 references. Required properties of engine antifreezes (alcohol, glycol, salt-base, petroleum, etc.) with comments on corrosion aspects in each case. Separate sections dealing with cooling-system corrosion, depletion of corrosion inhibitors and properties of coolant materials. —BNF. 12783

4.4.7

Additives in Steel Mill Lubricants. M. S. CLARK. The Texas Company. *Iron Steel Engr.*, **33**, No. 10, 87-91; disc., 91-93 (1956) Oct.

Concise description of various additives used in steel mill lubricants, their purpose and effects. Viscosity index improvers, extreme pressure agents, pour point depressants, detergents, dispersants, anti-corrosives, anti-oxidants, anti-foam agents, polar materials, soaps in greases, solvents, bactericides, rust inhibitors and solids are covered. Tables.—INCO. 13068

4.5 Soil

4.5.1

Electrochemical Aspects of Underground Corrosion and Cathodic Protection. W. E. EDWARDS. *Elec. Eng.*, **75**, No. 10, 907-912 (1956) Oct.

Discussion of basic electrochemical principles involved in corrosion of underground metallic structures. Table gives electrode potential series of common elements. Prevention of underground corrosion by protective coatings and cathodic protection is discussed.—INCO. 13004

4.5.1

Coefficient of Temperature and Heat of Activation of Corrosion Reactions of Iron in Soil. (In German.) T. MARKOVIC, M. KARSULIN, Z. DUGI AND D. ZAGAR. *Werkstoffe u. Korrosion*, **7**, No. 3, 138-139 (1956) March.

Corrosion of soft iron in different soils can be followed in relation to "soil-water/soil-air ratio" by means of coefficients of temperature and heats of activation of corrosion reactions.—BL. 12822

4.5.2, 6.3.6

Corrosion of Bronze in Sandy Soil. A Contribution to Research in Corrosion. (In German.) W. GEILMANN. *Angew. Chem.*, **68**, No. 6, 201-211 (1956) Mar. 21.

Disintegration of bronze in humous sandy soil is followed by analysis of a number of prehistoric bronzes. Rate is based on oxygen and carbon dioxide contents of ground waters. Tables, micrographs, photographs. 12 references.—BTR. 12008

4.5.3

Development of the Redox Probe. CARL G. DEUBER AND GEORGE B. DEUBER. AGA Research Project PM-20, April, 1956, 23 pp. Available from American Gas Association, 420 Lexington Avenue, New York, New York.

Development of the instrument devised by Starkey and Wight to measure the oxidation-reduction potential of soil is described. Aim of the project is to develop a rugged and reliable instrument which will permit quick establishment of the redox potential of soils.

The improved probe has been submitted to the gas industry for field testing. The book describes the theory and method of use of the probe. 13072

5. PREVENTIVE MEASURES

5.2 Cathodic Protection

Cathodic Protection in Action. J. P. BARRETT AND E. D. GOULD. *World Oil*, **142**, 149-153 (1956) March.

Cathodic protection today includes method of determining the need for cathodic protection; current requirements for its application; results of protection of casing; and cost of the process. Photographs, diagrams, graphs. 4 references.—BTR. 11933

5.3 Metallic Coatings

5.3.4, 6.4.2

Adherence and Corrosion of Electroplated Aluminium. F. SOLAR. *Products Finishing (U. S. A.)*, **18**, No. 8, 68-82 (even pages), (1954) May.

A free critical translation by S. Senderoff of the paper published in *Metalloberflache, B*, **5**, No. 8, 117-120 (1953) Aug. 9562

5.9 Surface Treatment

5.9.1

Surface Preparation of Steel. Pts. I, II. J. BIGOS. Paper before Montreal Corrosion Symp., Sept. 15-16, 1955. *Chem. Canada,*

J. M. PEARSON

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8, Nos. 5, 6, 30-34, 66-70 (1956) May, June.

Correct surface preparation is the primary and most important requirement for satisfactory painting of steel. Complete descaling by blast cleaning or pickling and a suitable pretreatment is the optimum surface preparation. For mild atmospheres, wire brushing is adequate and economical since intact millscale under such conditions is a good basis for paint. Various methods used in the preperation and pretreatment of steel, including solvent cleaning, steam cleaning, acid cleaning, chipping, wirebrushing, flame cleaning and sand blasting are outlined. Photos, tables.—INCO. 12370

7. EQUIPMENT

7.1 Engines, Bearings and Turbines

7.1

A Review of Some Combustion Problems Associated with the Aero Gas Turbines. J. S. CLARKE. *J. Roy. Aeronaut. Soc.*, **60,** No. 544, 221-240 (1956) April.

Pages 234-237 refer to the choice of materials for flame tube operating conditions. The original austenitic steel has been replaced successively by Inconel and Nimonic 75. Trouble is experienced from thermal fatigue resulting from repeated acceleration and deceleration of the engine and thermal fatigue data obtained for Nimonic 75 and other materials on a laboratory test rig given. A promising material is Nimoply—an inner layer of copper protected by outer layers of Nimonic 75.—BNF. 11948

7.1

The Development of Metal-Bonded Carbon Bearings. H. B. NUDELMAN, C. H. SUMP AND W. C. TROY. *ASTM Bull.*, No. 213, 62-69 (1956) April.

Development of a water-lubricated bearing material capable of performing in water at temperatures over 200 F. Study was made of methods of combining and bonding carbon and metals. Solder, 70-30 nickel-copper and 70-30 cobalt-chromium were metals used. Very effective bearings were made with the cobalt-chromium alloy, as revealed by water-lubricated sleeve-journal and thrust bearing tests. Impact tests of nickel-copper-graphite composites showed that compact containing 40 by volume of graphite was superior in impact strength to plain graphite. Sleeve-journal tests of 58 graphite-42 nickel-copper (70-30) showed that bearings containing fine carbon seized immediately while bearings with coarse graphite particles wore rapidly at light loads. Nickel-copper matrix provided predicted corrosion resistance, but failed to prevent welding. Tables, graphs, photomicrographs. 15 references.—INCO. 12095

7.4 Heat Exchangers

7.4.2, 2.2.3

How to Lengthen Condenser-Tube Life. H. A. TODHUNTER. *Power,* **100,** No. 3, 85-87 (1956) March.

Test installations of various types of condenser tubes at the Harbor Steam Plant showed that 70-30 copper-nickel tubes exhibited the least pitting of any of the tubes tested. After 30,000 hours of service, pits were less than 0.005-inch deep for copper-nickel with 0.4-0.6 iron. Other materials tested were aluminum-brass, Admiralty, phosphorized and arsenical Admiralty, aluminum-bronze and arsenical aluminum-bronze. Galvanic action may affect aluminum-bronze tubes installed alongside copper-nickel tubes. Only aluminum brass condenser tubes were in use while harbor water was polluted with hydrogen sulfide and 70-30 copper-nickel tubes now standardized for the plant may not be satisfactory if harbor water becomes polluted again. Other heat exchanger applications for copper-nickel tubing include 70-30 copper-nickel tubes in feedwater heaters, 80-20 copper-nickel steam tubing in the evaporators and 70-30 in salt water heat exchangers. Graphs.—INCO. 12135

7.5 Containers

7.5.2, 5.3.2, 2.3.2

Steel Surface Properties Affect Internal Corrosion Performance of Tin Plate Containers. A. R. WILLEY, J. L. KRICKL AND R. R. HARTWELL. *Corrosion,* **12,** No. 9, 433t-440t (1956) September.

Two easily-performed laboratory tests have been developed which give results correlating with internal corrosion performance of plain electrolytic tin plate containers. One, the "rate of pickling" test measures a permanent surface property of the steel arising in the annealing operation. The other, the "iron solution" test, measures the effect of certain factors governing the nature of the steel surface prior to electrotinning and to some extent the effect of the plating operation itself.

Tin plate manufacturers have used these tests as guides in improving the corrosion resistance of their product. The described tests, however, are not entirely adequate to account for all of the existing variations in corrosion performance believed to be related to steel surface characteristics. Additional work is required. 12143

7.6 Unit Process Equipment

7.6.4, 4.6.2, 2.3.1

Modern Analytical Techniques Control Corrosion of Boiler Steel. R. T. HANLON. *Modern Power & Eng.,* **50,** No. 3, 82-87 (1956) March.

Prevention of internal boiler deposits at elevated pressures and high heat transfer rates depends on effective control of feedwater quality and boiler water composition. Most failures of boiler metal are caused by overheating which results in changes in microstructure of steel. Modern laboratory techniques using precise chemical analyses, spectrographic analyses, X-ray diffraction, the petrographic microscope and the camera microscope have made it possible to specifically relate deposit composition to boiler and metal failure, whether it is on the fire side or water side of boiler and to ascertain degree of overheating to which tube metal has been subjected.—INCO. 12005

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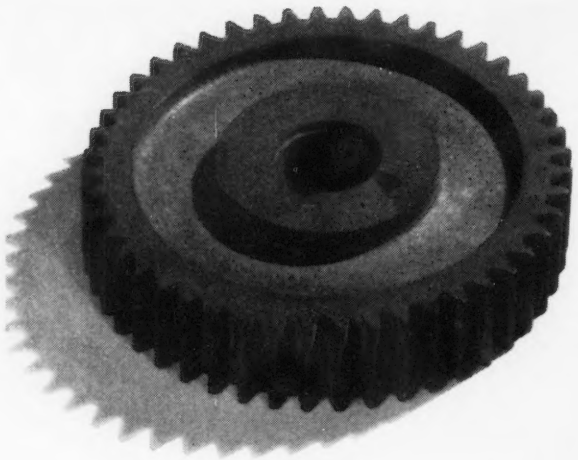

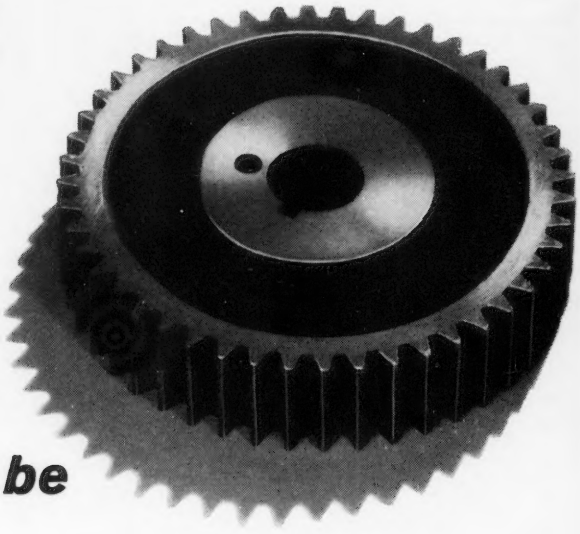

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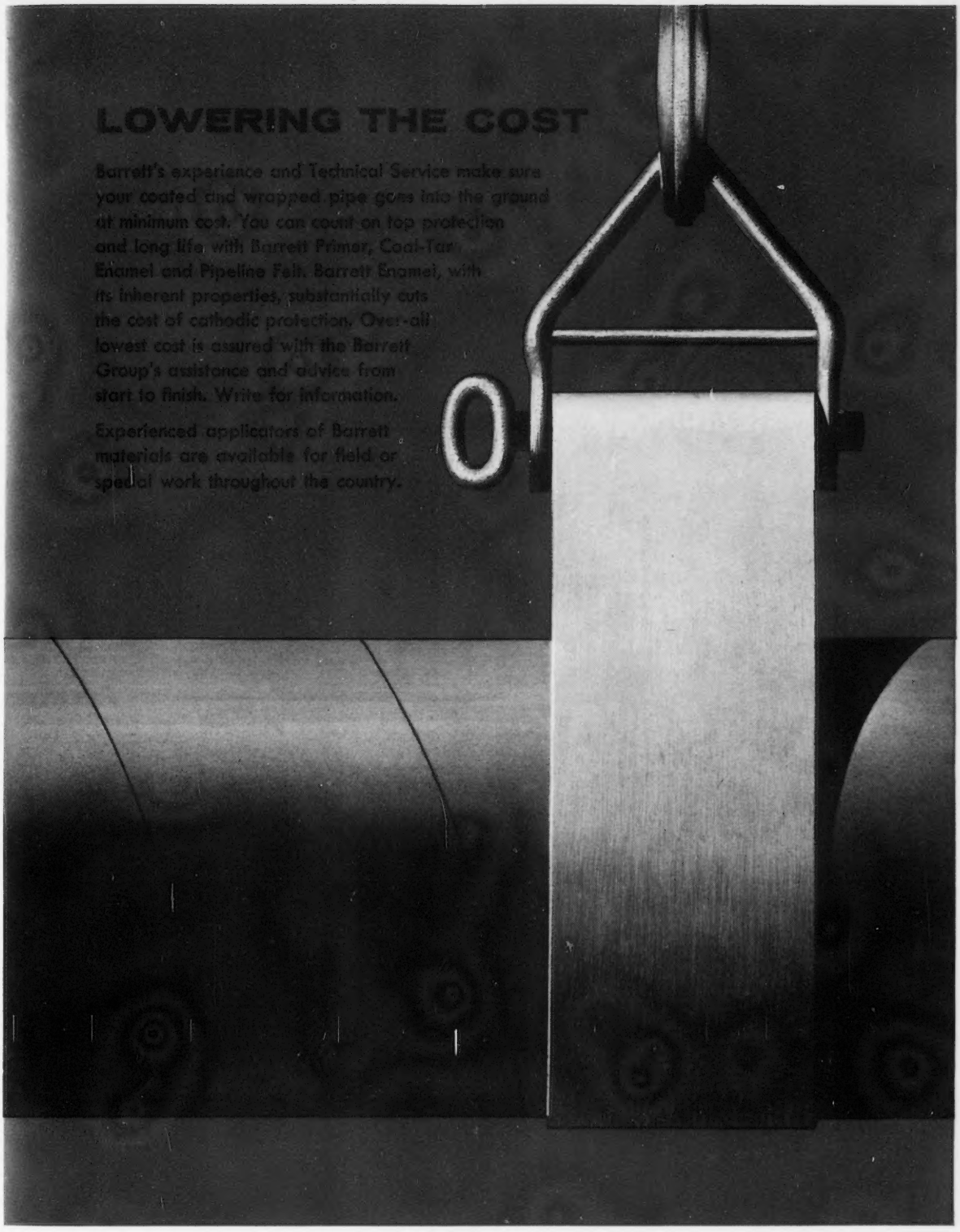
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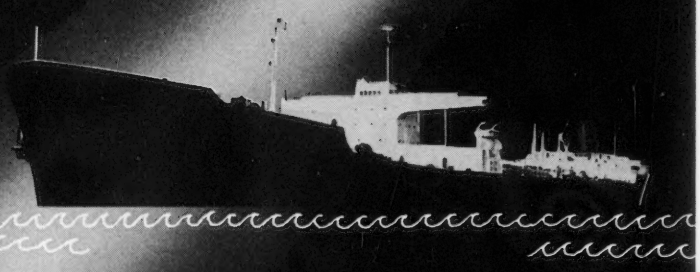

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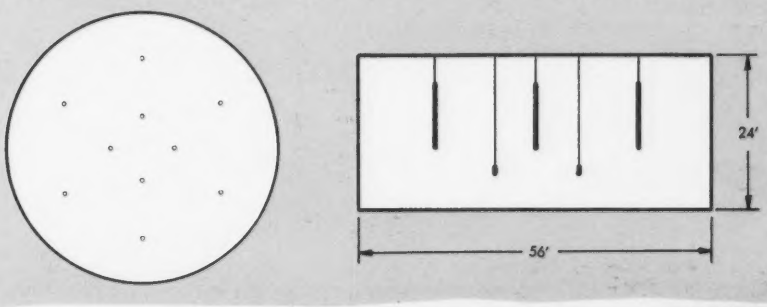

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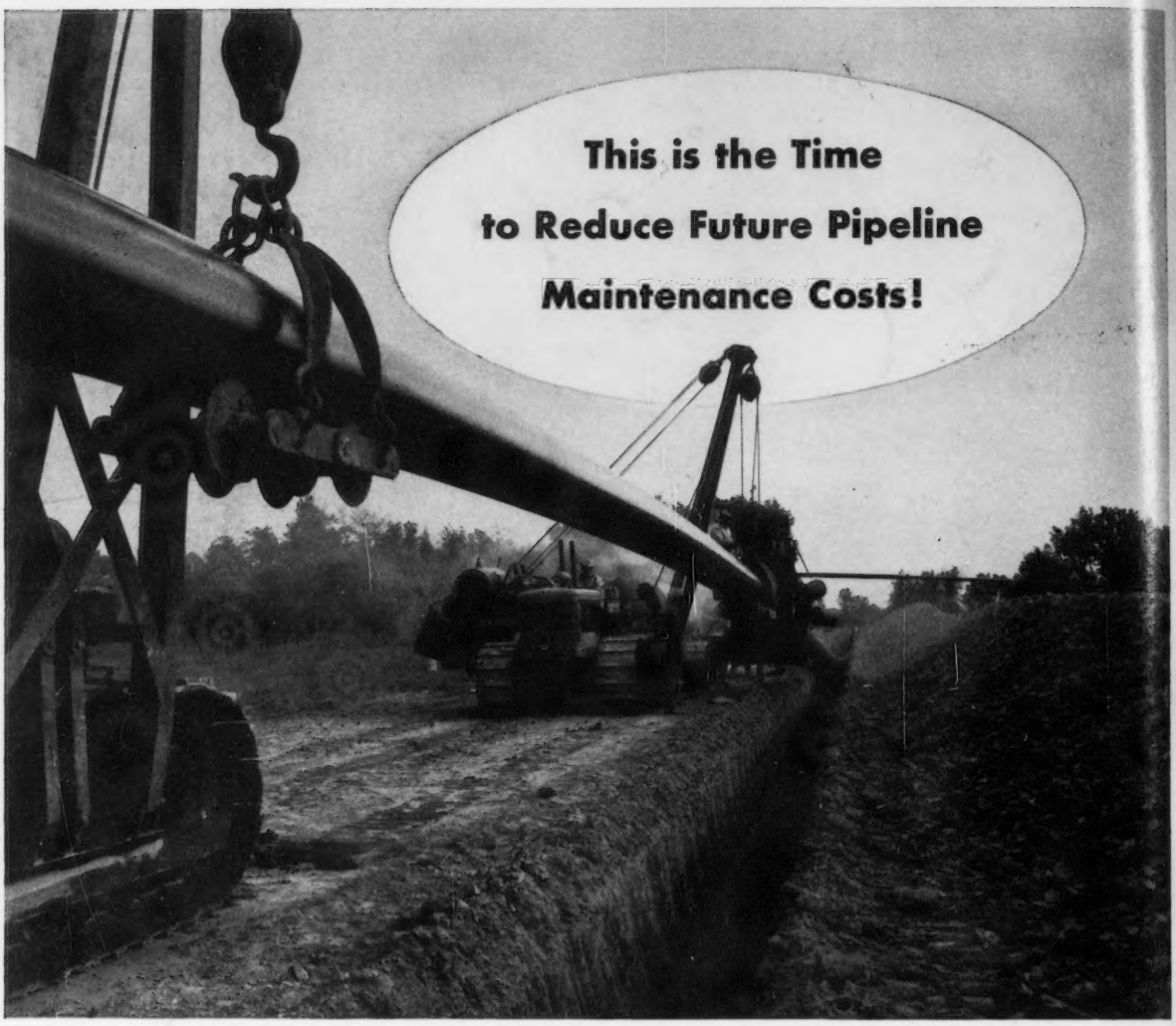

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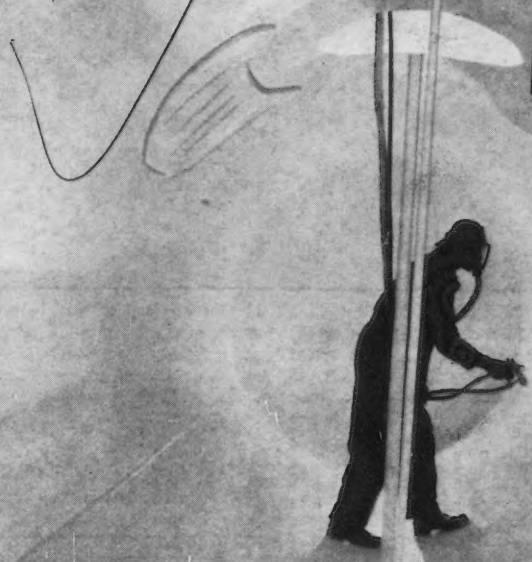

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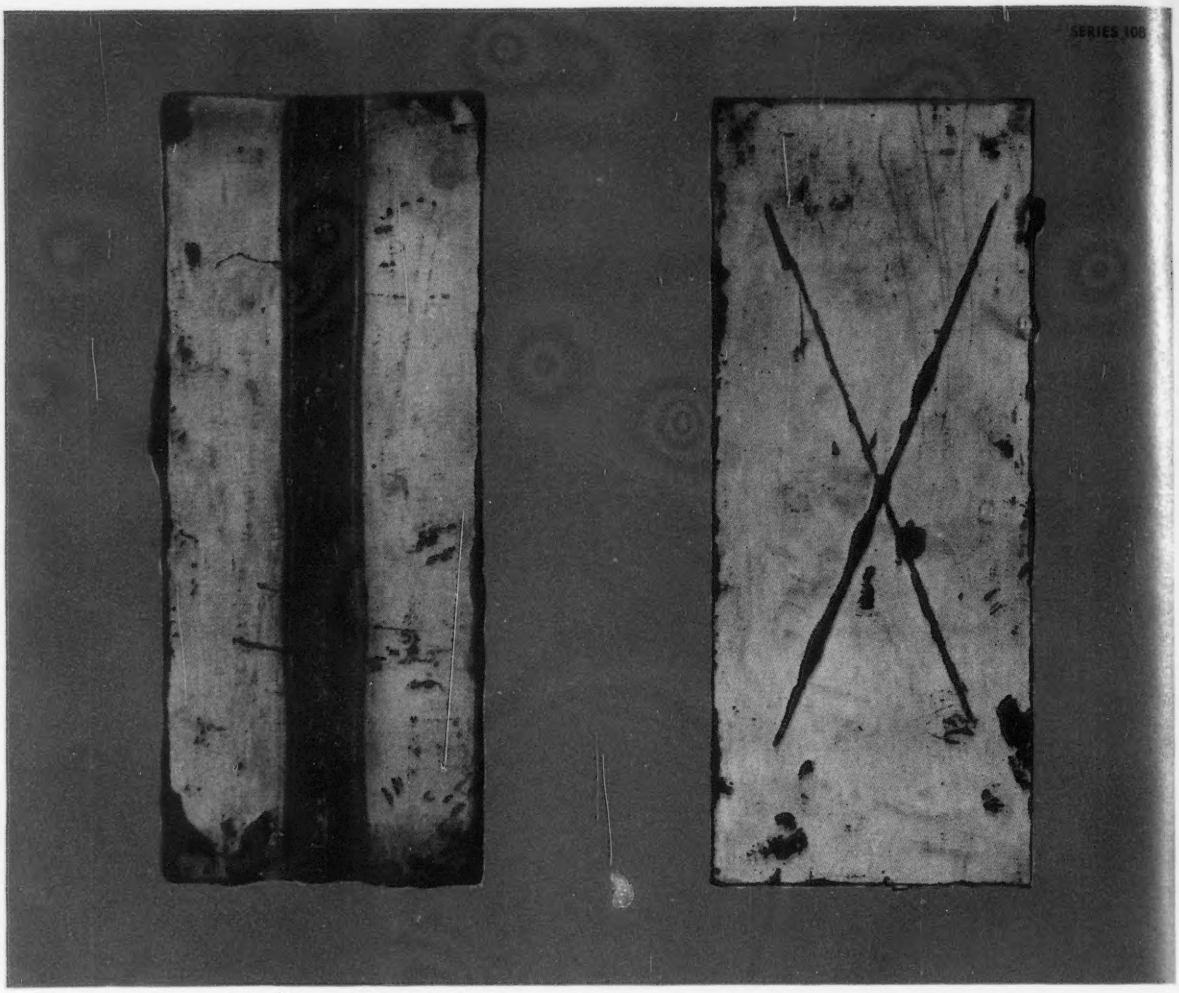

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